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June 14, 2010

**REMEDIAL INVESTIGATION  
FOR OPERABLE UNIT 3  
LIBBY ASBESTOS SUPERFUND SITE**

**PHASE IV SAMPLING AND ANALYSIS PLAN  
PART A – DATA TO SUPPORT  
HUMAN HEALTH RISK ASSESSMENT**

**Prepared by  
U.S. Environmental Protection Agency  
Region 8  
Denver, CO**



**With Technical Assistance from:**

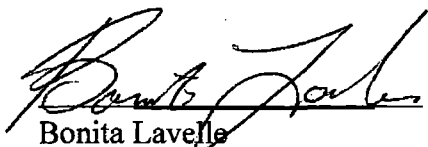
**SRC, Inc.  
Denver, CO**



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**APPROVAL PAGE**

This Phase IV Part A Sampling and Analysis Plan for Operable Unit 3 of the Libby Asbestos Superfund Site is approved for implementation.



Bonita Lavelle

Remedial Project Manager, Libby OU3

6/14/10  
Date

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**DOCUMENT REVISION LOG**

Revision	Date	Primary Changes
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## LIST OF ACRONYMS

ABS	Activity-Based Sampling
AOC	Administrative Order on Consent
ATV	All Terrain Vehicle
CAR	Corrective Action Request
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
COC	Chain-of-Custody
CSM	Conceptual Site Model
DQO	Data Quality Objective
EDD	Electronic Data Deliverable
EDXA	Energy Dispersive X-Ray Analysis
EPA	U.S. Environmental Protection Agency
FS	Feasibility Study
FSRZ	Fire Suppression Restricted Zone
FSDS	Field Sample Data Sheets
FSP	Field Sampling Plan
FTP	File Transfer Protocol
GIS	Geographic Information System
GO	Grid Opening
GPS	Global Positioning System
GSD	Geometric Standard Deviation
ID	Identification number
IL	Inter-laboratory
ISO	International Organization for Standardization
IUR	Inhalation Unit Risk
KDC	Kootenai Development Corporation
LA	Libby Amphibole
MCE	Mixed Cellulose Ester
MDEQ	Montana Department of Environmental Quality
MWH	MWH Americas, Inc
NVLAP	National Voluntary Laboratory Accreditation Program
OU	Operable Unit
PCM	Phase Contrast Microscopy
PCME	Phase Contrast Microscopy Equivalent
PDF	Portable Document Format
PLM	Polarized Light Microscopy
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QC	Quality Control

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RBC	Risk-Based Concentration
RBF	Risk-Based Fraction
RD	Recount Different
RI	Remedial Investigation
RPM	Remedial Project Manager
RS	Recount Same
SAED	Selective Area Electron Diffraction
SAP	Sampling and Analysis Plan
SOP	Standard Operating Procedure
TEM	Transmission Electron Microscopy
TWF	Time-Weighting Factor
UCL	Upper Confidence Limit
USFS	U.S. Forest Service

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**REMEDIAL INVESTIGATION  
FOR OPERABLE UNIT 3  
LIBBY ASBESTOS SUPERFUND SITE**

**PHASE IV SAMPLING AND ANALYSIS PLAN  
PART A – DATA TO SUPPORT  
HUMAN HEALTH RISK ASSESSMENT**

**1.0 PROJECT OVERVIEW**

**1.1 Purpose of This Document**

This document is the Sampling and Analysis Plan (SAP) for Phase IV Part A (Data to Support Human Health Risk Assessment) of the Remedial Investigation (RI) for Operable Unit 3 (OU3) of the Libby Asbestos Superfund Site (the site). This SAP contains the elements required for both a field sampling plan (FSP) and quality assurance project plan (QAPP), and has been developed in accordance with the U.S. Environmental Protection Agency (EPA) Requirements for Quality Assurance Project Plans (EPA 2001) and the Guidance on Systematic Planning Using the Data Quality Objectives Process – EPA QA/G4 (EPA 2006). The SAP is organized as follows:

- Section 1 – Project Overview
- Section 2 – Background and Problem Definition
- Section 3 – Data Needed For Human Health Risk Assessment
- Section 4 – Data Quality Objectives
- Section 5 – Sampling Program
- Section 6 – Laboratory Analysis Requirements
- Section 7 – Quality Control
- Section 8 – Sample Handling & Documentation
- Section 9 – Data Management
- Section 10 – Assessment and Oversight
- Section 11 – Data Validation and Usability
- Section 12 – References

**1.2 Project Management and Organization**

Project Management

EPA is the lead regulatory agency for Superfund activities within OU3. The EPA Remedial Project Manager (RPM) for OU3 is Bonita Lavelle, EPA Region 8. Ms. Lavelle is a principal data user and decision-maker for Superfund activities within OU3.

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The Montana Department of Environmental Quality (MDEQ) is the support regulatory agency for Superfund activities within OU3. The MDEQ Project Manager for OU3 is Dick Sloan. EPA will consult with MDEQ as provided for by the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA), the National Contingency Plan, and applicable guidance in conducting Superfund activities within OU3.

EPA has entered into an Administrative Order on Consent (AOC) with Respondents W.R. Grace & Co.-Conn. and Kootenai Development Corporation (KDC) for performance of a Remedial Investigation/Feasibility Study (RI/FS) at OU3 of the Libby Asbestos Site. Under the terms of the AOC, W.R. Grace & Co.-Conn. and KDC will implement this SAP. The designated Project Coordinator for Respondents W.R. Grace & Co.-Conn. and KDC is Robert Medler of Remedium Group, Inc.

### Technical Support

EPA will be supported in this Phase IV RI by a number of contractors, including:

- SRC, Inc. will assist in the development of SAPs, and in the evaluation and interpretation of the data.
- Formation Environmental, Inc., a contractor to SRC, will provide support in planning sampling and analysis activities, preparation of maps and other geographic information system (GIS) applications needed to summarize and interpret data, maintenance of a web site with site data, and evaluation of the feasibility study.
- HDR will provide oversight of field sampling and data collection activities.
- The U.S. Department of Transportation, John A. Volpe National Transportation Systems Center will implement the laboratory quality assurance (QA) program for OU3 and provide technical support.

### Field Sampling Activities

All field sampling activities described in this SAP will be performed by W.R. Grace & Co.-Conn. and KDC, in strict accord with the sampling plans developed by EPA. W.R. Grace & Co.-Conn. and KDC will be supported in this field work by MWH Americas, Inc. (MWH) and by their subcontractor Chapman Construction, Inc. Individuals responsible for implementation of field sampling activities are listed below:

- Project Manager: John Garr
- Field Team Leader: Toby Leeson
- Field Quality Control Officer: Stephanie Boehnke
- Quality Control Officer: Mike DeDen

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### On-Site Field Coordinator

Although most of the work described in this Phase IV SAP will be conducted on property owned by the US Forest Service (USFS), crews may need to access areas of OU3 via Rainy Creek Road. Access to the mine via Rainy Creek Road is currently restricted and is controlled by EPA. The on-site point of contact for access to the mine is Rob Burton of PRI:

Rob.burton@priworld.com  
406-293-3690

### Sample Preparation and Analysis

All samples collected as part of the Phase IV investigation will be sent for preparation and/or analysis at laboratories selected and approved by EPA. Laboratories that will be utilized for analysis of Phase IV asbestos samples may include Hygeia and EMSL.

### Data Management

Administration of the master database for OU3 will be performed by EPA contractors (SRC and Formation Environmental). The primary database administrator will be Lynn Woodbury of SRC. She will be responsible for sample tracking, uploading new data, performing data verification and error checks to identify incorrect, inconsistent or missing data, and ensuring that all questionable data are checked and corrected as needed. When the OU3 database has been populated, checked and validated, relevant asbestos data will be transferred into a Libby Asbestos Site database as directed by EPA for final storage.



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## **2.0 BACKGROUND AND PROBLEM DEFINITION**

### **2.1 Site Description**

Libby is a community in northwestern Montana that is located near a large open-pit vermiculite mine. Vermiculite from the mine at Libby is known to be contaminated with amphibole asbestos that includes several different mineralogical classifications, including richterite, winchite, actinolite and tremolite. For the purposes of EPA investigations at the Libby Asbestos Superfund Site, this mixture is referred to as Libby Amphibole (LA).

Historic mining, milling, and processing of vermiculite at the site are known to have caused releases of vermiculite and LA to the environment. Inhalation of LA associated with the vermiculite is known to have caused a range of adverse health effects in exposed humans, including workers at the mine and processing facilities (Amandus and Wheeler 1987, McDonald et al. 1986, McDonald et al. 2004, Sullivan 2007, Rohs et al. 2007), as well as residents of Libby (Peipins et al. 2003). Based on these adverse effects, EPA listed the Libby Asbestos Site on the National Priorities List in October 2002.

Starting in 2000, EPA began taking a range of cleanup actions at the site to eliminate sources of LA exposure to area residents and workers using CERCLA (or Superfund) authority. Given the size and complexity of the Libby Asbestos Site, EPA designated a number of Operable Units (OUs). This document focuses on investigations at OU3. OU3 includes the property in and around the former vermiculite mine and the geographic area surrounding the mine that has been impacted by releases and subsequent migration of hazardous substances and/or pollutants or contaminants from the mine, including ponds, Rainy Creek, Carney Creek, Fleetwood Creek, and the Kootenai River. Rainy Creek Road is also included in OU3.

Figure 2-1 shows the location of the mine and a preliminary study area boundary for OU3. EPA established the preliminary study area boundary for the purpose of planning and developing the scope of the remedial investigation/feasibility study (RI/FS) for OU3. This study area boundary may be revised as data are obtained during the RI for OU3 on the nature and extent of environmental contamination associated with releases that may have occurred from the mine site. The final boundary of OU3 will be defined by the final EPA-approved RI/FS.

### **2.2 Basis for Concern**

EPA is concerned with environmental contamination in OU3 because the area is used by humans for logging, a variety of recreational activities, and in the case of USFS employees, land management and fire fighting activities. The area is also habitat for a wide range of ecological receptors (both aquatic and terrestrial). Contaminants of potential concern to EPA in OU3

include not only LA, but any other mining-related contaminants that may have been released to the environment.

### **2.3 Scope and Strategy of the RI at OU3**

As noted above, Respondents W.R. Grace & Co.- Conn. and KDC are performing an RI in OU3 under EPA oversight in order to characterize the nature and extent of environmental contamination and to collect data to allow EPA to evaluate risks to humans and ecological receptors from mining-related contaminants in the environment.

The RI is being performed in several phases. Phase I of the RI was performed in the fall of 2007 in accord with the *Phase I Sampling and Analysis Plan for Operable Unit 3* (EPA 2007). The primary goal of the Phase I investigation was to obtain preliminary data on the levels and spatial distribution of asbestos and non-asbestos contaminants that might have been released to the environment in the past as a consequence of the mining and milling activities at the site.

Phase II of the OU3 RI was performed in the spring, summer, and fall of 2008. Phase II was composed of three parts, as follows:

- Part A (EPA 2008a) focused on the collection of data on the levels of LA and other chemicals of concern in surface water and sediment, as well as site-specific toxicity testing of surface water using rainbow trout.
- Part B (EPA 2008b) focused on the collection of data on LA levels in ambient air samples collected near the mined area, and on the collection of data on LA and other chemicals of potential concern in groundwater.
- Part C (EPA 2008c) focused on the collection of other data needed to support the ecological risk assessment at the site.

Phase III of the RI was performed primarily in the spring, summer, and fall of 2009, with some activities (e.g., fish toxicity testing, geotechnical studies) still ongoing. The details of the plan are provided in EPA (2009a). Phase III included the collection of activity-based air samples during simulated recreational visitor activities in the forested area, as well as the collection of a variety of ecological community and habitat metrics in support of the ecological risk assessment, toxicity testing of surface water using rainbow trout, and toxicity testing of surface water and sediment using amphibians.

### **2.4 Scope and Purpose of the Phase IV SAP**

The Phase IV SAP describes the sampling and analysis that will be performed during Phase IV of the OU3 RI. This document (Phase IV, Part A) describes the activities planned to collect data to support the human health risk assessment. The details of data collection in support of the ecological risk assessment will be provided in Part B of the Phase IV SAP.

### 3.0 DATA NEEDED FOR HUMAN HEALTH RISK ASSESSMENT

#### 3.1 Conceptual Model for Human Exposure to Asbestos

Figure 3-1 presents a conceptual site model (CSM) for human exposure to asbestos that summarizes EPA's current understanding of the environmental media in OU3 that are likely to be contaminated by past and ongoing releases of LA from the mine, and the pathways by which humans might be exposed to LA, now or in the future. The CSM for LA focuses on pathways of inhalation exposures, because the inhalation pathway is generally considered to be of much greater risk than oral or dermal pathways for human exposure. The CSM has been revised since the Phase III SAP was implemented based on EPA's revised understanding of human activities that are reasonably expected to occur within OU3.

A range of different human receptors may be exposed to LA in OU3, including:

- *Trespasser or "rockhound" in the mined area* – This population includes older children and adults who trespass on the area that has been disturbed by past mining activities. In this document, this is referred to as the "mined area". Exposures of potential concern for asbestos include inhalation of ambient air and inhalation of air in the vicinity of soil, duff, and solid waste (e.g., tailings, ore) disturbances.
- *Recreational visitors in the forested area* – This receptor population includes older children (assumed to be age 7 or older) and adults who engage in activities such as camping, hiking, dirt bike riding, all terrain vehicle (ATV) riding, hunting, etc. Exposures of primary concern for asbestos include inhalation of ambient air, inhalation of air in the vicinity of contaminated soil, duff (organic debris), or roadways/trails disturbed by recreational activity, and inhalation of LA released from contaminated tree bark while gathering wood for a campfire and while burning the wood in a campfire.
- *Recreational visitors along streams and ponds* – This receptor population includes adults and older children who hike, fish, wade/swim or explore site drainages. In the absence of access restrictions, this might include the streams and ponds along Fleetwood Creek, Carney Creek, and Rainy Creek, as well as reaches of the Kootenai River that may be impacted by site releases. Exposures of potential concern for asbestos include inhalation of ambient air and inhalation of air in the vicinity of duff, dried soils or sediments that are disturbed by walking or exploring drainages. As noted above, exposure from ingestion of LA in fish is judged to be of minor concern compared to inhalation exposures that would occur during visits to OU3.

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- *Residential wood harvester in the forested area* – This receptor population includes adult area residents who engage in sawing, hauling, and stacking wood for personal use. Exposures of potential concern for asbestos in OU3 include inhalation of ambient air, inhalation of airborne emissions of LA from roadways and inhalation of air that contains LA released from soil or duff as well as LA fibers released to air by cutting and hauling timber that has LA in the tree bark.
- *Commercial loggers in the forested area* – This receptor population includes adult workers who are employed in commercial logging operations in OU3. Exposures of potential concern for asbestos include inhalation of ambient air, inhalation of airborne emissions of LA from roadways and inhalation of air that contains LA released from soil or duff as well as LA fibers released to air by cutting and stacking timber that has LA in the tree bark.
- *Forest service workers in the forested area* – This population includes employees of the U.S. Forest Service (USFS) who may engage in a range of forest management activities, including maintenance of roads and trails, cutting fire breaks, thinning and trimming trees, measuring trees, etc. Exposures of potential concern for asbestos include inhalation of ambient air, inhalation of airborne emissions of LA from roadways and inhalation of LA released to air from management activities that disturb soil, tree bark or duff.
- *Forest service fire fighters in the forested area* – This population includes employees of the USFS who respond to forest fires that occur within OU3. For ground-based fire fighters, exposures of potential concern include inhalation of ambient air, inhalation of LA released to air from disturbance of soil, duff, and tree bark while performing activities such as cutting fire lines and , as well as inhalation of LA released to smoke by the fire. For pilots of aircraft that respond to fires in OU3, the exposure of concern is inhalation of LA that is released to smoke and that enters the aircraft as it passes through the smoke column.
- *Area residents* – Area residents who do not enter OU3 are not likely to be exposed to LA from OU3 except via inhalation exposure to LA released into air during a forest fire.

Note that other residential exposure scenarios are not included in the CSM for OU3 because any properties geographically within OU3 that are currently residential will be evaluated for routine residential scenarios as part of OU4. Based on information currently available to EPA, future residential development is not reasonably anticipated in other areas of OU3.

### Pathways Selected for Quantitative Investigation in Phase IV

Not all of the exposure scenarios to asbestos identified in Figure 3-1 are of equal concern or require equal levels of investigation. The following sections identify the pathways of chief

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concern to EPA and which are considered to warrant quantitative evaluation in the human health risk assessment.

### *Exposure to Ambient Air*

All people who are present in OU3 may be exposed to LA in ambient air. Therefore, this pathway is selected for quantitative evaluation. Data to support the evaluation of exposure to ambient air was collected in the Phase I and Phase II RI.

### *Exposures of Trespasser/Rockhound within the Mined Area*

The mined area is characterized by the occurrence of vermiculite interspersed with veins of LA exposed by mining, as well as large piles of mine waste, waste rock, and a coarse tailings pile. Sampling results from the Phase I remedial investigation at OU3 indicate that levels of LA greater than 1% occur at multiple locations in the mined area. The Phase I sampling results, along with observations of veins of LA exposed by mining, provide sufficient information to conclude that sources present are very likely to be of concern to human health. EPA guidance contained in OSWER Directive 9200.0-68 ("Framework for Investigating Asbestos-Contaminated Superfund Sites", EPA 2008d), provides that "if data indicate high levels of asbestos are present in soil (e.g., >1%), a risk manager may determine that a response action should be undertaken and that further efforts to characterize the source or potential airborne exposures before action is taken are not needed." Therefore, EPA has concluded that response action is necessary to prevent human exposure to LA within the mined area of OU3. EPA anticipates that access restrictions to the mined area and adjacent lands surrounding the mined area that are owned by KDC (including the unpaved portion of Rainy Creek Road) will be part of an OU3 response action and that quantification of hypothetical future exposures of trespassers within this mined area and surrounding W.R. Grace-owned property is not needed to support risk management decision-making. EPA expects that alternatives to prevent human access to the mined area will be evaluated in the feasibility study for OU3.

### *Exposures of Recreational Visitors in the Forest Area*

Recreational visitors who enter the forested area around the mine site may be exposed to asbestos during a wide variety of activities that disturb contaminated source media, including soil, duff, and tree bark. The reasonable maximum exposure includes:

- Inhalation exposure while walking or hiking
- Inhalation exposure while riding an ATV
- Inhalation exposure while actively disturbing soil or duff when clearing a campsite or building a fire
- Inhalation exposure when gathering wood with LA contamination in bark for a campfire
- Inhalation exposure to smoke from burning wood with contaminated bark in a campfire.

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All of these activities are considered to be plausible and potentially important in evaluating human exposure in OU3, so all of these activities are selected for quantitative evaluation. Note that due to the steep terrain, camping occurs mainly on roadways or at the sides of roadways within OU3. Data to support the evaluation of these activities was collected in the Phase III RI.

### *Exposures of Recreational Visitors Along Ponds and Creeks*

Sediments in ponds and creeks that drain OU3 are known to be contaminated with LA, and recreational visitors who disturb the sediments while walking or fishing along the ponds or creeks might be exposed to LA released to air. In this regard, release of LA from sediments that are submerged is not of concern, and release from sediments that are exposed but still wet is likely to be relatively low. However, releases from contaminated sediments that become exposed and dry out during periods of low water could be of concern. These activities are selected for quantitative evaluation. Data to support the evaluation of these activities will be collected in Phase IV RI, Part A.

Since EPA anticipates that access restrictions to the mined area and adjacent lands owned by KDC will be part of an OU3 response action, data collection to support evaluation of recreational visitors along ponds and creeks will focus on the lower portion of Rainy Creek which is outside the boundary of KDC-owned property.

### *Exposure of Commercial Loggers*

The best approach for characterizing human exposure during this activity would be to monitor air levels during authentic commercial logging activities near the site. EPA will consider the need to investigate this scenario in the future after consideration of the results for the other scenarios that will be evaluated.

### *Exposures of USFS Workers*

USFS workers have the potential to be exposed to LA released from disturbed soil, duff and tree bark during a range of different forest management activities such as trail maintenance, tree thinning, and stand examination. These activities are selected for quantitative evaluation, and data to support the evaluation of these activities will be collected in Phase IV RI, Part A.

### *Exposures of USFS Workers and Firefighters*

USFS firefighters have the potential to be exposed to LA released from disturbed soil, duff and tree bark when responding to wildfires in OU3. For ground-based firefighters, exposures of chief concern include cutting firelines by hand and with heavy equipment. For pilots who respond by air, the exposure of chief concern is inhalation of LA in smoke that enters the aircraft

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cockpit. These activities are selected for quantitative evaluation, and data to support the evaluation of these activities will be collected in Phase IV RI, Part A.

### *Exposures of Area Residents*

Area residents who harvest wood for use as a heating source have the potential to be exposed to LA released from disturbed soil, duff, and tree bark. During a forest fire within OU3, area residents also have the potential to be exposed to LA released in smoke from burning trees. Data to support the evaluation of these activities will be collected in Phase IV RI, Part A.

### **3.2 Data Needs for Human Health Risk Assessment**

As discussed in EPA (2008d), evaluation of risk to humans from inhalation exposure to asbestos requires reliable estimates of the long-term average concentration of asbestos in breathing zone air. At present, it is not possible to reliably calculate breathing zone air concentrations based only on knowledge of asbestos levels in source materials (soil, duff, tree bark, etc.), so the best approach is usually to obtain multiple direct measurements of asbestos in air for use in the risk assessment. This is generally referred to as Activity-Based Sampling (ABS), where the activities may range from passive (little or no disturbance of contaminated source materials) to a range of active disturbances of source materials. EPA guidance (2008d) recommends focusing on active disturbances of source materials to support Superfund risk management decisions.

To date, ABS data have been collected for the recreational visitor in forest area (during the Phase III RI). EPA believes that the ABS data collected for recreational visitors in the forest area during the Phase III RI adequately characterize some (e.g., inhalation of LA from disturbance of soil and duff while hiking/walking) but not all of the exposure pathways for the additional populations of potential concern, so it is concluded that additional ABS data are needed to support exposure and risk evaluations for other populations.

Therefore, the Phase IV investigation will seek to obtain adequate ABS data for five of the six populations of concern in OU3 for which no ABS data currently exist:

- Recreational visitors along lower Rainy Creek
- Residential wood harvesters
- USFS workers
- USFS firefighters
- Area residents exposed to smoke from a forest fire

The potential need to perform ABS studies to represent exposures to commercial loggers in OU3 will be considered in the future, after review and evaluation of the Phase IV ABS data.



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#### **4.0 DATA QUALITY OBJECTIVES FOR PHASE IV ABS DATA COLLECTION**

EPA has developed a seven-step process for establishing data quality objectives (DQOs) to help ensure that data collected during a field sampling program will be adequate to support reliable site-specific risk management decision-making (EPA 2001, 2006). These seven steps are presented below.

##### **4.1 Step 1: State the Problem**

The Phase I remedial investigation for OU3 of the Libby Asbestos Site included collection of data on levels of LA in tree bark, duff, and forest soils within the Kootenai National Forest surrounding the mined area. The Phase I data indicate that LA was detected by polarized light microscopy (PLM) in soils at distances up to 2 miles from the mine in the downwind direction. LA was detected by transmission electron microscopy (TEM) in samples of tree bark and duff in downwind, cross wind and upwind directions at distances from 3 to 7.5 miles from the mine. There is general tendency for the highest levels detected in tree bark and duff samples to occur within about 2 to 3 miles of the mined area. It's currently unknown whether the detected LA presents an unacceptable risk to human health.

As stated in the Framework for Investigating Asbestos-Contaminated Superfund Sites (EPA 2008d), asbestos fibers in source material are typically not inherently hazardous, unless the asbestos is released from the source material into air where it can be inhaled. If inhaled, asbestos fibers can increase the risk of developing lung cancer, mesothelioma, pleural fibrosis, and asbestosis.

EPA will perform an assessment of risk to human health from exposure to LA released from tree bark, soil and/or duff within the OU3 study area in order to decide whether remedial action is warranted and where. Evaluating risks to humans from exposure to asbestos is most reliably achieved by collection of data on the level of asbestos in breathing zone air during disturbance of a source of asbestos (i.e., ABS sampling) (EPA 2008d). Information on the level of LA in breathing zone air released from disturbed tree bark, soil, and duff is needed to complete a risk assessment for OU3. However, at present, there are no ABS data that are adequate to evaluate the exposures of recreational visitor along streams and ponds, residential wood harvesters in OU3, USFS workers and firefighters in OU3, or area residents exposed to smoke from fires in OU3.

##### **4.2 Step 2: Identify the Goal of the Study**

The goal of the Phase IV RI is to provide sufficient data to allow EPA to complete an exposure assessment for recreational visitors along streams and ponds, residential wood harvesters, USFS workers and firefighters in OU3 and area residents exposed to smoke from fires in OU3. EPA

will use the exposure assessment in an evaluation of risks to human health. The risk assessment will support decisions about whether or not response actions are needed to protect various sub-populations of humans from unacceptable risks from LA in air that is attributable to releases from human disturbances of contaminated environmental media in OU3 and releases resulting from fires.

#### 4.3 Step 3: Identify Information Inputs

The information needed to characterize human exposures from activities in OU3 consists of reliable and representative measurements of LA concentrations in air under exposure scenarios that are characteristic of the activities engaged in by members of each of the populations of people described above. Such measurements are obtained by drawing a known volume of air through a filter that is located in the breathing zone of the individual performing the disturbance activity and measuring the number of LA fibers that become deposited on the filter surface.

The information needed to characterize exposure of area residents to LA in smoke from forest fires consist of reliable and representative measurements of LA in ambient air during a fire in the area of the forest impacted by LA.

#### 4.4 Step 4: Define the Bounds of the Study

*Spatial Bounds:* The spatial bounds of the study include the OU3 study area identified in Figure 2-1. For each sub-population, exact sampling locations should include areas that are representative of the scenario being evaluated, as follows:

- Recreational visitors along streams and ponds. As noted above, EPA anticipates that access to land owned by W.R Grace and Co. will be restricted as part of a response action in OU3. This includes the area that encloses the tailings impoundment, the Mill pond, and Carney and Fleetwood Creeks. However, the lower portion of Rainy Creek below the boundary of the W. R. Grace-owned property may be open to recreational visitors. Based on this, the area of chief concern for this population is along lower Rainy Creek, from the W.R. Grace property line to the Kootenai River.
- Residential wood harvesters. This population of people may be exposed at any location within OU3 where wood harvesting is permitted by the USFS. Based on this, ABS sampling for the residential wood harvester scenario should occur at multiple areas within OU3. Available data on levels of LA measured in tree bark, soil and duff indicate that, in general, the levels of LA tend to decrease with distance away from the center of the mine. Since wood harvesting could occur anywhere within the forested area of OU3, ABS sampling to characterize exposures for the residential wood harvester should occur in areas where relatively high, average, and low levels of LA have been detected in tree bark, soil and duff.

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- USFS workers. This population of people may be exposed at almost any location within OU3. Based on this, ABS sampling for the USFS worker populations should occur at multiple areas within OU3. ABS sampling to characterize exposures for USFS workers should occur in areas where relatively high, average, and low levels of LA have been detected in tree bark, soil and duff.
- USFS fire fighters. Based mainly on available data on LA levels in soil, duff and tree bark, the USFS has established a "fire suppression restricted zone" (FSRZ) around the mined area within which fire fighting is restricted to aerial attack. This FSRZ is the area that is of chief concern. ABS data are needed to evaluate the risk associated with fighting fires in this area using ground crews to determine the need for the FSRZ and if needed, the boundary. Also, ABS data are needed to evaluate the risk associated with fighting fires in this area using aerial attack. ABS sampling to characterize exposures for USFS fire fighters should occur in areas of relatively high, average, and low levels of LA that have been detected in tree bark, soil, and duff within the FSRZ.
- Area residents exposed to smoke. Area residents may be exposed to smoke from forest fires mainly in the community of Libby including residential areas around the perimeter of OU3. EPA (2009b) established three monitoring stations to measure LA in air during significant fire events, including a) the CDM office building in Libby, b) the campground east of OU3 at McGillivray Access, and c) the USFS ranger station along Highway 37. These three stations are reasonable and representative, and will be maintained for use in Phase IV. During a significant fire event, one additional mobile station will be established downwind of the fire.

*Temporal Bounds:* The release of LA from source materials (dried sediment, soil, duff, tree bark) into air is expected to depend on several factors that may tend to vary over time, including, for example, the moisture content of the source, the amount of ground cover, and the wind speed and direction when disturbance occurs. Therefore, ABS data should, to the extent practicable, be collected over a sufficient time frame to ensure the data are representative of the long-term mean concentration level. In general, it is expected that human exposures are more likely to occur when snow is limited or absent from OU3, and that releases will tend to be higher in the dry summer months than during wet conditions in spring or fall. Based on this, most of the ABS sampling events should occur in the time frame of June-October. To avoid collecting data that are biased low, ABS sampling should not occur during or within 1 day of rainfall ( $>1/4$  inch). This approach will help ensure that the mean concentration calculated using the set of measurements obtained during dry periods is more likely to overestimate than underestimate the actual long term mean exposure.

### 4.5 Step 5: Define the Analytical Approach

The results of the ABS program in OU3 will be used to calculate an exposure point concentration for each population at each ABS location. The exposure point concentration will be the average air concentration measured over multiple rounds of sampling. Note that each round of ABS may

require the collection of several air samples collected during separate disturbance activities to avoid overloading the filters. In such cases, the exposure point concentration will be the average of all samples and all rounds of sampling. The data on ambient air concentrations during a forest fire will be used to calculate an exposure point concentration for residents.

The exposure point concentration will be combined with assumptions about exposure frequency and duration for each scenario and toxicity factors for LA in a baseline risk assessment for OU3 that is expected to provide a basis for EPA to determine, in consultation with MDEQ, whether response action is needed within OU3 to protect human health. EPA guidance contained in OSWER Directive 9355.0-30, "Role of the Baseline Risk Assessment in Superfund Remedy Selection Decisions" (EPA 1991) indicates that where the cumulative carcinogenic risk to an individual based on reasonable maximum exposure for both current and future land use is less than  $1E-04$  and the non-carcinogenic hazard quotient is less than 1, remedial action is generally not warranted unless there are adverse environmental impacts. The guidance also states that a risk manager may decide that a risk level lower than  $1E-04$  is unacceptable and that remedial action is warranted where there are uncertainties in the risk assessment results.

Human health risk will be calculated for each scenario at each ABS location. Ideally, there would be sufficient ABS locations to allow risks to be calculated at various distances from the mine and in all directions. However, this would require ABS sampling over an area of over 100 square miles. The approach that will be taken is to collect ABS samples in the predominantly downwind direction from the mine and to assume that the risks calculated at these locations are equal to or greater than the risks at equal distances from the mine in the crosswind and upwind directions. This approach will help ensure that assumed risks at locations in the up- and cross-wind directions are more likely to be overestimated than underestimated. If deemed to be needed to support risk management decisions, additional ABS at locations in the cross- and up-wind directions may be added in the future. Any additional sampling locations will be specified in a modification to this SAP.

#### **4.6 Step 6: Specify Performance Criteria**

In making decisions about the risks to humans in OU3, two types of decision errors are possible:

1. A false negative decision error would occur if a risk manager decides that exposure to LA in OU3 is not of health concern, when in fact it is of concern.
2. A false positive decision error would occur if a risk manager decides that exposure to LA in OU3 is above a level of concern, when in fact it is not.

EPA is most concerned about guarding against the occurrence of false negative decision errors, since an error of this type may leave humans exposed to unacceptable levels of LA in OU3. EPA guidance recommends that because of the uncertainty in estimating the true average concentration within an exposure area, the 95% upper confidence limit of the arithmetic mean

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(95UCL) should be used when calculating exposure and risk to humans. The 95UCL provides reasonable confidence that the true average will not be underestimated and controls false negative decision errors. In general, as the number of samples increases, uncertainties decrease, the value of the 95UCL moves closer to the true mean and exposure evaluations using either the mean or the 95UCL produce similar results. For this reason, it is anticipated that risk management decisions at OU3 will be based not only on the best estimate of the long-term average concentration at each ABS sampling area, but will also consider an estimate of the 95UCL at each ABS sampling area. Use of the 95 UCL to estimate exposure and risk at each exposure area helps account for limitations in the data, and provides a margin of safety in the risk calculations, ensuring that risk estimates are more likely to overestimate than underestimate the true risk level.

EPA is also concerned with the probability of making false positive decision errors. Although this type of decision error does not result in unacceptable human exposure, it may result in unnecessary expenditure of resources. For the purposes of this planning effort, the strategy adopted for controlling false positive decision errors is to seek to ensure that, if the risk estimate based on the best estimate of the mean is  $\leq \frac{1}{2}$  the level of concern but the estimate based on the 95UCL is above EPA's level of concern, then the ratio of the risk estimates (risk based on the 95UCL divided by risk based on best estimate of the mean) is less than a factor of 3. For example, if the risk estimate based on the mean were 10% of the level of concern and the risk estimate based on the 95UCL were 50% of the level of concern (a ratio of 5), the data would be considered to adequate for decision-making. However, if the risk estimate based on the mean were 40% of the level of concern and the risk estimate based on the 95UCL were twice (200%) the level of concern (also a ratio of 5), then it would be concluded that there is a substantial probability of a false positive error and that more data may be needed to strengthen decision-making. Conversely, if the risk estimate based on the mean were 80% of the level of concern and the risk estimate based on the 95UCL were twice the level of concern (a ratio of 2.5), then it would be concluded that there is only a small probability of a false positive error and that collection of additional data would be unlikely to improve the basis for decision-making.

### **4.7 Step 7: Develop the Plan for Obtaining Data**

#### *Activities to be Included in the ABS*

For each sub-population to be evaluated in Phase IV, there are a variety of different activities that might result in exposure to LA. Because the focus is on collecting data that are representative of the long-term average, the ABS scenario for each of the sub-populations will include activities that are considered to be realistic and representative for the population being assessed. These scenarios are described in "scripts" that are implemented by individuals who collect the ABS data. These scripts specify the types of activity to engage in, and the relative length of time for each activity. These scripts are provided in Attachment A.

### *Selection of Sampling Locations*

Because of the very complex nature of the source material (a mixture of duff, soil, and tree bark), the difficulty in thoroughly characterizing the LA concentrations in these source media, and the potential difficulty in establishing a reliable quantitative relation between source and ABS air, no attempt will be made to establish a quantitative relation between LA levels in source media and the mean concentration in ABS air. Rather, ABS air data will be collected at multiple locations in OU3, selected to be representative of locations where the scenario of concern is likely to occur. This in turn yields information on the spatial pattern of exposure and risk.

The strategy for selection of sampling locations is based mainly on a consideration of spatial representativeness, and is also informed by available data on LA levels in source media (soil, duff and tree bark) as a function of distance and direction from the mined area. These data, collected along seven transects radiating from the mined area during the Phase I investigation, are summarized in Figure 4-1A (soil), Figure 4-1B (bark), Figure 4-1C (duff) and Figure 4-1D (all three combined).

Figure 4-2 shows candidate ABS "study areas" for use during the Phase IV ABS program.

- For the recreational visitor along Rainy Creek, the ABS study area extends from the W.R. Grace-owned property south to the Kootenai River. This area is evaluated as a single unit since it is not large enough to support multiple locations.
- For the residential wood harvester, three study areas will be studied. These shall be in the primary downwind direction (north-northeast) from the mine site. Each area shall be accessible by truck, and shall contain at least 5 trees within 50 yards of the road that are suitable for harvesting (this designation is made by the USFS). Tentative locations are shown in Figure 4-2. These areas will be referred to as "Near" (Area 10), "Middle" (Area 7), and "Far" (Area 2).
- For most of the USFS management worker and the USFS firefighter scenarios, the same three study areas (Near, Middle, and Far) used for wood harvesting shall be used. As above, these three areas are selected to provide spatial representativeness as a function of distance, which is expected to be correlated with the level of LA contamination in environmental media. However, one scenario (fighting a fire with heavy equipment) can not be safely done within these three areas. Rather, the heavy equipment scenario will be performed at locations that are safe for this activity, as indicated by the three areas with red cross-hatching in Figure 4-2.
- For exposure to smoke during simulated wildfires, two large slash piles that exist in OU3 are identified as the most appropriate locations for simulated wildfires (see Figures 4-3 and 4-4). These slash piles are located approximately at the 5-mile point on the mine haul road, about 100 yards on the northern side of the tailings pile.
- The sampling locations for ambient air monitors to be activated in the event of a forest fire in the FSRZ of OU3 are: 1) the CDM office building in Libby; 2) the USFS Canoe Gulch

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Ranger Station on Highway 37; 3) the McGillivray Campground on the west shore of Lake Koocanusa; and 4) a mobile monitor to be located downwind of the fire.

In the event that any changes in sample locations are needed for reasons of safety or implementability, the revisions in the locations of study areas will be specified in a modification to this SAP.

### *Optimizing Sample Number*

As discussed in Step 6 of the DQO process for the Phase IV study, one data quality objective is to limit false positive decision errors such that, if the risk associated with the mean of a data set is  $< \frac{1}{2}$  the level of concern but the risk estimate based on the 95UCL is above EPA's level of concern, then the ratio of the 95UCL to the mean should not exceed a factor of about 3. As discussed in EPA (2009a), the ratio of the 95UCL to the true mean depends on a large number of factors, the most important of which are the number of samples and the degree of between-sample variability. If the between-sample variability is low (e.g., geometric standard deviation [GSD]  $\leq 3$ ), then the number of samples needed to ensure the risk estimate based on the 95UCL is within a factor about 3 of the risk estimate based on the mean is estimated to be 10 to 15. However, if the GSD is larger, then the number of samples needed is likely on the order of at least 25 to 50, depending on the size of the GSD.

At present, data are not available to estimate how close the mean concentration of LA in ABS air is to a level of human health concern for the scenarios to be evaluated in Phase IV, or on the magnitude of the underlying variability. In the absence of such data, the minimum number of samples to be collected and analyzed in this effort for each scenario is 10 per ABS area per script. This should be sufficient to support decision making at each area if the between-sample variability is not too large and if the observed mean concentration is not too close to a decision threshold. Additional sampling may be needed to support decision-making in cases where the data are variable and/or are near a decision threshold.

In order to minimize health and safety concerns, a different approach will be taken to evaluate exposure of USFS fire fighters to LA released in smoke from burning trees and duff. As described in Attachment A, exposure to smoke from a wildfire will be evaluated by collecting personal air samples during the burning of two large slash piles in OU3. During each fire, 4 individual samplers will collect 2 samples each for a total of 8 personal air samples per fire (16 total).

For the characterization of exposure of area residents to smoke from a forest fire, samples of ambient air will be collected every time a fire of significant size occurs within the FSRZ. The number of samples this will generate is unknown.



### *Selection of Target Analytical Sensitivity*

The level of analytical sensitivity needed to ensure that analysis of ABS air samples from OU3 will be adequate is derived by finding the concentration of LA in ABS air that might be of potential concern, and then ensuring that if an ABS sample were encountered that had a true concentration equal to that level of concern, it would be quantified with reasonable accuracy.

At present, EPA has not developed a quantitative procedure for evaluating non-cancer risks associated with inhalation exposure to asbestos, but has developed a method for quantification of cancer risk (EPA 2008d). The basic equation is:

$$\text{Risk} = C \cdot \text{TWF} \cdot \text{IUR}_{a,d}$$

where:

- C = Average concentration of asbestos structures in inhaled air (s/cc)
- TWF = Time weighting factor to account for less than continuous exposure (unitless)
- IUR<sub>a,d</sub> = Inhalation unit risk (s/cc)<sup>-1</sup> based on continuous exposure beginning at age “a” and continuing for duration “d” years. EPA (2008d) provides a table (Table E-4) of unit risk values for a range of start ages and exposure durations.

It is important to recognize that the value of C must be expressed in units of Phase Contrast Microscopy (PCM) f/cc. This is because the current risk model for estimation of cancer risk from inhalation exposure to asbestos (EPA 2008d) is based on cumulative exposure expressed as PCM f/cc-yrs. The concentration of PCM fibers in ABS air could be measured directly using phase contrast microscopy, but EPA believes it is better to measure the concentration of total LA fibers using TEM, and then to compute the number of fibers observed in TEM that meet the counting requirements for PCM<sup>1</sup>. These are referred to as PCM-equivalent (PCME) fibers. The concentration of PCME fibers (measured by TEM) is an estimate of the concentration value that would have been obtained if the sample were analyzed by PCM. Since the number of PCME fibers released under the scenarios being evaluated under Phase IV is not yet known, for the purpose of determining target analytical sensitivity, the number of PCME fibers is based on the average ratio of PCME to total LA fibers measured in other samples collected from the site. This is referred to as the “risk-based fraction” (RBF), and the calculation is performed as follows:

$$C(\text{PCME}) = C(\text{total LA}) \cdot \text{RBF}_{\text{PCME}}$$

<sup>1</sup> In the PCM method, a fiber is counted if it has a length of 5 µm or longer and an aspect ratio of at least 3:1. Although there is no thickness rule, particles thinner than about 0.25 µm are not usually detectable by PCM. Hence, the counting rules for PCME are: length ≥ 5, aspect ratio ≥ 3, thickness > 0.25.

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Combining the equations above and re-arranging to solve for the concentration of LA that corresponds to a specified risk level yields the following:

$$C(\text{total LA}) = \text{Specified Risk} / [\text{RBF}_{\text{PCME}} \cdot \text{TWF} \cdot \text{IUR}_{\text{a,d}}]$$

For convenience, the concentration of LA that corresponds to a specified risk level is referred to as a Risk-Based Concentration (RBC).

Table 4-1 (Panel A) identifies the exposure parameters that have been selected to calculate the RBC for each of the exposure scenarios being investigated in Phase IV. These exposure parameters are intended to be conservative, which helps ensure that the target analytical sensitivity will be adequate. Exposure parameters used in the final human health risk assessment may be different than those used here.

Table 4-1 (Panel B) shows the calculation of the RBC for each exposure scenario. The value of the RBF is based on available data from previous ABS studies in OU3 indicated that 36 out of 71 total LA structures were PCME (RBF = 0.51). This value is similar to what has been observed in samples collected in studies in OU4. The target risk employed in these calculations is 1E-05 in all cases. It is important to emphasize that choice of 1E-05 as the "specified risk" is not a risk management decision about the need for remedial action. Rather, this choice is strictly for the purposes of deriving an analytical sensitivity that will be adequate for the OU3 Phase IV ABS program. All actual evaluations of health risk will be performed by EPA in the risk assessment for OU3, and all risk management decisions will be documented in the Record of Decision.

Table 4-1 (Panel C) calculates the target sensitivity for each scenario, based on the RBC values derived in Panel B. In all cases, the target sensitivity is set so that, on average, about 5 LA structures would be counted in a sample whose true concentration was equal to the RBC. This level of analytical sensitivity should be sufficient to allow reliable quantitation of ABS samples that approach or exceed a risk level of about 1E-05.

Table 4-1 (Panel D) shows the estimated number of grid opening that may be required to achieve the specified target sensitivity for each scenario.

### *Optimizing the Sample Collection Strategy*

Two key variables that may be adjusted during collection of air samples are sampling duration and pump flow rate. The product of these two variables determines the amount of air drawn through the filter, which in turn is an important factor in the analytical cost and feasibility of achieving the target analytical sensitivity (see above). In general, longer sampling times are preferred over shorter sampling times because a) longer time intervals are more likely to yield representative measures of the average concentration (as opposed to short-term fluctuations), and b) longer collection times are associated with higher volumes, which makes it easier to achieve

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the target analytical sensitivity. Likewise, higher flow rates are generally preferred over lower flow rates because high flow results in high volumes drawn through the filter over shorter sampling times.

However, there is a limit to how much air can be drawn through a filter. In cases where the air being sampled contains a significant level of dust, high air volumes may lead to overloading of the filter with dust particles. In this event, the filter cannot be examined directly, but must undergo an "indirect preparation" in which the material on the filter is suspended in water and only a fraction is re-deposited on a "secondary" filter, such that the secondary filter is not overloaded. In some cases, indirect preparation of air samples may alter (usually increase) the observed concentration of asbestos in air samples. Region 8 has reviewed published studies on this topic (see HEI-AR 1991 and Breysse 1991 for reviews), and interprets the data to indicate that, in contrast to what is usually observed in the case of chrysotile asbestos, effects of indirect preparation of samples containing amphibole asbestos are generally small (e.g., Bishop et al. 1978, Sahle and Laszlo 1996). However, because of the possibility that indirect preparations might cause changes (increases) in measured LA concentrations, EPA has determined that, for this project, it is desirable to seek to limit the volume of air drawn through the filter to an amount that approaches but does not cause overloading in order to minimize the need for use of indirect preparations.

Based on experience gained from implementation of the OU3 Phase III recreational visitor ABS sampling effort, it was found that most ABS samples were overloaded when collected using the originally planned sampling conditions (180 minutes at a flow of 8 L/minute). Based on this finding, the original ABS sampling protocol for the recreational visitor scenario (EPA 2009a) was revised to divide the composite activity into three sub-activities, and to reduce the sampling time and pump flow rate for each (EPA 2009c). Each individual wore two sampling pumps which operated at a flow rate of either 2 L/min (low flow) or 4 L/min (high flow). Whenever possible, the filter from the high flow pump was selected for analysis. In cases where the high flow filter was overloaded, then the low flow filter was analyzed. Average values for Phase III samples are as follows:

Activity	Average Time (min)	Average Flow (L/min)	Average Vol. (L)
ATV Riding	20	3.76	75
Hiking	80	3.00	240
Fire building	35	2.82	99

Based on this experience, the strategy for collecting Phase IV ABS samples shall be similar to that used in the revised Phase III approach (EPA 2009c), where each individual wears two pumps, operated at 2 L/min and 4 L/min. Sampling times should be adjusted so that the majority of the high flow filters approach but do not exceed overloading. In cases when overloading does occur, then the low flow sample should be analyzed.

## **5.0 SAMPLING PROGRAM**

Table 5-1 provides an overview of the Phase IV ABS sampling design. Key elements are discussed in greater detail below.

### **5.1 ABS Scripts**

As discussed above, individuals will engage in a timed series of different activities to generate ABS samples that are representative of a range of realistic activities that may be performed by each population being assessed. The scripts are presented in Attachment A.

### **5.2 Sampling Areas**

Sampling areas for ABS data collection and for smoke monitoring during naturally occurring wildfires are shown in Figure 4-2.

### **5.3 Sampling Schedule**

#### Recreational Visitor Along Rainy Creek Scenario

The recreational scenario along lower Rainy Creek will be implemented in middle to late summer (late July through early September) when flow in Rainy Creek is low, to maximize the chance of hikers being exposed to dried sediment. Samples should be collected on warm and dry days and should not be collected within 24 hours of a rain event.

#### Residential Wood Harvester Scenario

Similar to the recreational visitor, it is expected that wood harvesting by area residents will occur mainly when the weather is warm and snow cover is minimal or absent, with a majority of the activity occurring in late summer or early fall. Based on this, the residential wood harvester scenario will be implemented in middle to late summer (late July through early September) to optimize the conditions for releasing LA. Samples should be collected on warm and dry days and should not be collected within 24 hours of a rain event.

#### USFS Forest Management Scenarios

It is expected that most routine forest management activities performed by USFS staff in OU3 will occur when the weather is warm and snow cover is minimal or absent. Based on this, the time window for collection of ABS samples for the USFS forest management work is approximately June 1 through September 30.

#### Fireline Construction Scenarios

Exposure of USFS firefighters may occur at any time of year that a fire occurs. Based on USFS records, the highest frequency of fires in the Kootenai national Forest occurs in the months of

July, August, and September. Based on this, the time window for collecting data for the cutting of firelines (both by hand and using heavy equipment) will be between July 1 and September 30.

#### Simulated Wildfire Scenario

For exposure to smoke from simulated wildfires, it is vital that the fires occur under conditions when risk of the fire spreading is minimal. Consequently, these scenarios will be implemented under wet or snowy conditions. The choice of time for these events will be closely coordinated with the USFS and will be subject to USFS approval. On the day(s) selected for the simulated wildfires, one fire will be ignited in the morning, and the second fire will be ignited in the afternoon. This is because meteorological conditions often vary significantly between morning and afternoon, and this can influence the behavior of the smoke plume.

#### **5.4 Activity Patterns within Each Area**

In order to maximize the representativeness of the samples over space as well as time, to the extent feasible, the exact locations of the ABS activities within the ABS areas should vary from event to event. In order to create a record of the exact locations within each ABS area that were evaluated, each person will carry a global positioning system (GPS) unit programmed to automatically record location ( $\pm$  about 5 meters) once every minute. Field crews will download this electronic record at the end of each ABS event. The Field Quality Control Officer and the Field Team Leader will be responsible for ensuring that ABS events are conducted at different locations within the ABS area. Any questions about the representativeness of sampling locations will be directed to the EPA Remedial Project Manager (RPM) for resolution. At the completion of the Phase IV ABS program (all ABS events completed at all areas), the tracks from all ABS events at each ABS areas will be superimposed to create maps of the locations that were evaluated at each area across the entire sampling investigation. These maps shall be submitted to EPA and MDEQ.

#### **5.5 Personal Air Sampling Protocol**

All ABS air samples will be collected in accord with SOP ABS-LIBBY-OU3 (Rev. 0). A copy of this standard operating procedure (SOP) is presented in Attachment B. All air samples will be collected using cassettes that contain a 25 mm diameter mixed cellulose ester (MCE) filter with a pore size of 0.8  $\mu$ m. As discussed above, during initial sampling events, pumps will be set to a flow rate of either 2 L/min (low flow) or 4 L/min (high flow). Sampling durations are specified in the scripts for each ABS scenario (see Attachment A). These flow rates and sampling times may be revised as experience is gained on the degree of loading on the ABS filters.

A battery-powered air sampling pump (SKC model AirChek XR5000<sup>TM</sup> (0.005-5.0 L/min) or similar) will be worn by the participant. The monitoring cassette will be attached to the pump via a plastic tube, and affixed to the shoulder of the participant such that the cassette is within the breathing zone. The breathing zone can be visualized as a hemisphere approximately 6 to 9

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inches around an individual's face. The top cover from the cowl extension on the sampling cassette shall be removed ("open-face") and the cassette oriented face down.

Each air sampling pump will be calibrated at the start of each ABS sampling period using a rotameter that has been calibrated to a primary calibration source. For pre-sampling purposes, calibration will be considered complete when the measured flow is within  $\pm 5\%$  of the target flow, as determined by the mean of three measurements. Each rotameter used for field calibration will be transported to and from each sampling location in a sealed zip-top plastic bag. The cap used at the end of the rotameter tubing will be replaced each morning after it is used.

As noted in the ABS script (see Attachment A), the pumps should be turned on at the beginning of each ABS event, and left to run for the duration of the script or activity as specified. Because flow may tend to change during the ABS script, flow will also be measured and recorded at the completion of the script.

To prevent potential cross-contamination between different ABS sample collection events, individuals who perform sequential ABS sample collection activities will don clean personal protection equipment (PPE) before beginning each new event. Similarly, non-disposable equipment (ATVs, shovels, rotameters, etc.) will be decontaminated between ABS events at different locations in accord with OU3 SOP No. 7.

### **5.6 Collection of Bark Samples from Slash Piles**

Before ignition of simulated wildfires at either of the large slash piles located in OU3, it is necessary to collect bark samples from each slash pile in order to characterize the level of LA contamination in the pile.

Bark samples will be collected and prepared for analysis in basic accord with SOP TREE-LIBBY-OU3 (Rev. 1). Because the trees in the slash pile are no longer standing, samples should be collected about 4-5 feet above the cut base of trees that are approximately 8 inches or larger in diameter. A total of 8 bark samples shall be collected from each pile. These shall be collected from 8 different trees, located at representative random locations in the slash pile. Initially, 2 samples from each pile will be analyzed to verify the presence of LA in the tree bark. More samples may be analyzed if the initial results do not indicate the presence of LA.

### **5.7 Collection of Ambient Air Samples During Wildfires in OU3**

As noted above, EPA (2009b) established three monitoring stations to measure LA in air during significant fire events in OPU3, including a) the CDM office building in Libby, b) the campground east of OU3 at McGillivray Access, and c) the USFS ranger station along Highway 37. This plan has been amended to add a fourth monitoring location, downwind of the fire location. This revised plan is provided at Attachment D.

Note that these monitors may also be activated during any USFS controlled burns in OU3, and during simulated wildfires initiated by EPA as part of the OU3 RI, if wind conditions carry smoke from the fires towards Libby.

## **5.8 Field Documentation**

All data associated with each ABS event shall be recorded on a field sample data sheet (FSDS) specifically designed for ABS activities in OU3. These FSDS forms are provided in the respective OU3 SOPs.

## **6.0 LABORATORY ANALYSIS REQUIREMENTS**

### **6.1 Laboratory Qualifications**

All laboratories that analyze samples of ABS air or tree bark for asbestos as part of this project must participate in and have satisfied the certification requirements in the last two proficiency examinations from the National Institute of Standards and Technology/National Voluntary Laboratory Accreditation Program (NVLAP). Laboratories must also have demonstrated proficiency by successful analysis of Libby-specific performance evaluation samples and/or standard reference materials and must participate in the on-going laboratory QA program for the Libby OU3 project.

### **6.2 Analytical Method and Counting Rules**

All samples of air and bark collected during Phase IV sampling will be submitted for asbestos analysis using transmission electron microscopy (TEM) in accord with the International Organization for Standardization (ISO) 10312 method (ISO 1995) counting protocols, with all applicable Libby site-specific laboratory modifications, including the most recent versions of modifications LB-000016, LB-000019, LB-00028, LB-000030, LB-000066, and LB-000085 (see Attachment C). All amphibole structures (including not only LA but all other asbestos types as well) that have appropriate Selective Area Electron Diffraction (SAED) patterns and Energy Dispersive X-Ray Analysis (EDXA) spectra, and having length greater than or equal to 0.5  $\mu\text{m}$  and an aspect ratio (length:width)  $\geq 3:1$ , will be recorded on the Libby site-specific laboratory bench sheets and electronic data deliverable (EDD) spreadsheets. Data recording for chrysotile, if observed, is not required.

### **6.3 Stopping Rules**

The target analytical sensitivities for ABS samples for each scenario are shown in Table 4-1.

The target analytical sensitivity for tree bark samples is 10,000  $\text{cm}^{-2}$ .

For all ABS and tree bark field samples, evaluate each sample until one of the following is achieved:

- A minimum of 2 grid openings (GOs) in each of 2 grids has been examined.
- The target sensitivity is achieved.
- 50 LA structures are observed
- An area of 1.0  $\text{mm}^2$  has been examined (approximately 100 GOs)

When one of these goals is achieved, complete the final GO and stop.



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For all ABS blanks (i.e., lot blanks, field blanks, and lab blanks), evaluate an area of 0.1 mm<sup>2</sup> (approximately 10 GOs) and stop.

## **7.0 QUALITY CONTROL**

Quality Control (QC) consists of the collection of data that allow a quantitative evaluation of the accuracy and precision of the field data collected during the project. QC samples that will be collected during ABS sampling include both field-based and laboratory-based QC samples.

### **7.1 Field-Based Quality Control Samples**

#### *Lot Blanks*

Before any air cassettes may be used for asbestos sampling, the lot must be determined to be asbestos free. This will be accomplished by selecting 2 lot blanks at random from the group of cassettes to be used for collection of ABS air samples. Each lot blank will be submitted for TEM analysis as described above. Once the lot is confirmed to be asbestos free (i.e., both lot blanks are non-detect after evaluation of an area of  $0.1 \text{ mm}^2$ ), that lot may be placed into use for sampling.

#### *Field Blanks*

A field blank for air shall be prepared by removing the sampling cassette from the box, opening the cassette to the air in the area where the investigative samples will be taken, then closing the cassette and packaging for shipment and analysis. Field blanks for ABS air will be collected at a rate of 1 per ABS sampling round. The ABS sampling location where the field blank is generated should be selected at random, choosing a new location (ABS area) for each field blank. This strategy will generate a total of 10 field blanks.

A field blank for tree bark shall be prepared using wood chips obtained from a home improvement store located outside of Libby, MT. One field blank sample will be submitted with the tree bark field samples.

### **7.2 Laboratory-Based Quality Control Samples for Asbestos Analysis by TEM**

#### *Air*

The QC requirements for TEM analyses of air samples at the Libby site are patterned after the requirements set forth by NVLAP. There are three types of laboratory-based QC analyses that are performed for TEM. Each of these is described below.

*Lab Blank* - This is an analysis of a TEM grid that is prepared from a new, unused filter in the laboratory and is analyzed using the same procedure as used for field blank samples.

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*Recounts* - A recount is an analysis where TEM grid openings are re-examined after the initial examination. The type of recount depends upon who is performing the re-examination. A *Recount Same* (RS) describes a re-examination by the same microscopist who performed the initial examination. A *Recount Different* (RD) describes a re-examination by a different microscopist within the same laboratory than who performed the initial examination. An *Interlab* (IL) describes a re-examination by a different microscopist from a different laboratory.

*Repreparation* - A repreparation is an analysis of a TEM grid that is prepared from a new section of filter as was used to prepare the original grid(s). Typically, this is done within the same laboratory as did the original analysis, but a different laboratory may also prepare grids from a new piece of filter.

For this project, the frequency of these laboratory-based QC samples will be as follows:

QC Sample Type	QC Sample Rate	Estimated Number (a)
Lab Blank	1% (1 per 100)	3
Recount Different	2% (1 per 50)	6
Interlab	2% (1 per 50)	6
Repreparation	2% (1 per 50)	6

(a) Assumes approximately 300 ABS samples will be analyzed during Phase IV-A

The list of samples for Recount Different, Interlab, and Repreparation will be selected by SRC and provided to the laboratory by the EPA RPM after the results of the original sample analyses have become available.

The most recent version of laboratory modification LB-000029 (see Attachment C) summarizes the acceptance criteria and corrective actions for TEM laboratory QC analyses that will be used to assess data quality.

### Tree Bark

There are two types of laboratory-based QC analyses for tree bark that will be performed as part of this study. Each is described below.

*Lab Blank* - A laboratory blank is a filter that is prepared by processing a clean crucible in the same way that a tree bark sample is prepared. A clean crucible is placed in the oven (with the sample set) at the same time that tree bark field samples are undergoing ashing. After ashing, the blank crucible is treated in the same manner as the field samples, and a filter is prepared for TEM examination. This type of blank is intended to

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indicate if contamination is occurring at any stage of the sample preparation procedure. One lab blank will be analyzed.

*Repreparation* - A repreparation is an analysis of a TEM grid that is prepared from a new section of filter as was used to prepare the original grid(s). Typically, this is done within the same laboratory as did the original analysis, but a different laboratory may also prepare grids from a new piece of filter. One repreparation will be analyzed. The most recent version of laboratory modification LB-000029 (see Attachment C) summarizes the selection process, acceptance criteria, and corrective actions for repreparations.

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## **8.0 SAMPLE HANDLING AND DOCUMENTATION**

### **8.1 Field Procedures**

#### **8.1.1 Sample Documentation and Identification**

Data regarding each sample collected as part of Phase IV sampling activities will be documented in accord with OU3 SOP No. 9 using Libby OU3-specific FSDS forms. At the time of collection, each sample will be labeled with a unique 5-digit sequential identification (ID) number. The sample IDs for all samples collected as part of Phase IV sampling activities will have a prefix of "P4" (e.g., P4-12345), unless specified otherwise. Information on whether the sample is representative of a field sample or a field-based QC sample (e.g., field blank, field duplicate/split) will be documented on the FSDS.

Each field sampling team will maintain a field log book. The log book shall record all potentially relevant information on sampling activities and conditions that are not otherwise captured on the FSDS forms. Examples of the type of information to be captured in the field log include:

- Names of team members
- Current and previous weather conditions
- Field sketches
- Physical description of the location relative to permanent landmarks
- Number and type of samples collected
- Any special circumstances that influenced sample collection
- Any deviations from sampling SOPs
- For ABS samples, the location description (what trails and areas) the ABS activities were performed in

As necessary for sample collection and location documentation, photographs will be taken using a digital camera. GPS coordinates will be recorded for all sampling locations on the FSDS form. A flag, stake or pole identifying the sampling station will be placed at or near the location for future identification.

#### **8.1.2 Handling Filter Cassettes**

All filter cassettes collected during the Phase IV-A effort will be handled as specified in SOP ABS-LIBBY-OU3 (Rev. 0).

#### **8.1.3 Holding Times**

There are no holding time requirements for the analysis of asbestos.

#### 8.1.4 Chain of Custody and Shipment

Field sample custody and documentation will follow the requirements described in OU3 SOP No. 9. Sample packaging and shipping will follow the requirements described in OU3 SOP No. 8.

A chain-of-custody (COC) form specific to the Libby OU3 sampling shall accompany every shipment of samples to the analytical laboratory. The purposes of the COC form are: a) to establish the documentation necessary to trace possession from the time of collection to final disposal, and b) to identify the type of analysis requested. All corrections to the COC record will be initialed and dated by the person making the corrections. Each COC form will include signatures of the appropriate individuals indicated on the form. The originals will accompany the samples to the laboratory and copies documenting each custody change will be recorded and kept on file. One copy of the COC form will be kept by field personnel.

All required paper work, including sample container labels, COC forms, custody seals and shipping forms will be fully completed in indelible ink (or printed from a computer) prior to shipping of the samples to the laboratory. Shipping to the appropriate laboratory from the field or sample storage will occur through overnight delivery.

All samples that may require special handling by laboratory personnel to prevent potential exposure to LA or other hazardous substances will be clearly labeled.

### 8.2 **Laboratory Procedures**

#### 8.2.1 Chain of Custody

Upon sample receipt, the laboratories will implement the following procedures:

- A sample custodian will be designated.
- Each sample shipment will be inspected by the sample custodian to assess the condition of the shipping container and the individual samples. The enclosed COC form will be reviewed and cross-referenced with all the samples in the shipment. Any discrepancies or abnormalities in samples will be noted and the EPA Project Manager or the appropriate delegate will be promptly notified. The EPA Project Manager shall be notified by telephone at (303) 312-6579 or email at [lavelle.bonita@epa.gov](mailto:lavelle.bonita@epa.gov).
- The COC form will be signed by the sample custodian and placed in the project file.
- Sample storage will be secured in the appropriate environment (i.e., refrigerated, dry, etc.), sample storage records and intra-laboratory sample custody records will be maintained, and sample disposal and disposal date will be properly documented.

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- Internal COC procedures will be followed by logging and assigning a unique laboratory sample number to each sample upon receipt (this number identifies the sample through all further handling at the laboratory).
- Internal logbooks and records will maintain the COC throughout sample preparation, analysis, and data reporting. These records will be kept in the project files.
- The original COC form will be returned to the Project QA Officer with the resulting data report from the laboratory.

Chain-of-custody will be maintained until final disposition of the samples by the laboratory and acceptance of analytical results.

### 8.2.2 Documentation and Records

Data reports will be submitted to EPA's technical contractor (SRC) in accordance with the procedures described in Section 8.2.3 below. Data reports shall include a case narrative that briefly describes the number of samples, the analyses, and any analytical difficulties or QA/QC issues associated with the submitted samples. The data report will also include signed COC forms, analytical data summary report pages, and a summary of laboratory QC sample results and raw data, where applicable. Raw data are to consist of instrument preparation and calibration logs, instrument printouts of field sample results, laboratory QC sample results, calibration and maintenance records, COC check in and tracking, raw data count sheets, spectra, micrographic photos, and diffraction patterns.

### 8.2.3 Data Deliverables

Asbestos data generated during this project will be entered into Libby-specific EDD spreadsheets by appropriately trained data entry staff. The data will include all relevant field information regarding each environmental sample collected, as well as the analytical results provided by the laboratory. Analytical results will include the structure-specific data for all TEM analyses. All data entry will be reviewed and validated for accuracy by the laboratory data entry manager or appointed delegate.

All asbestos EDDs will be submitted to EPA's technical contractor (SRC) electronically. Whenever possible, data files should be transmitted by e-mail to the following address:

[LibbyOU3@srcinc.com](mailto:LibbyOU3@srcinc.com)

When files are too large to transmit by e-mail, they should be provided on compact disk to the following address:

Lynn Woodbury  
SRC, Inc.



999 18th Street, Suite 1975  
Denver CO 80202  
(303) 357-3127

All original data records (both hard copy and electronic) will be cataloged and stored in their original form until otherwise directed by the Project Manager. At the termination of Phase IV, all original data records will be provided to the EPA Project Manager in a format specified by EPA for incorporation into the OU3 project files.

#### 8.2.4 Archival and Final Disposition

All sample materials, including filters, grids, and cassettes will be maintained in storage at the laboratory unless otherwise directed by EPA. When authorized by EPA, the laboratory will be responsible for proper disposal of any remaining samples, sample containers, shipping containers, and packing materials in accordance with sound environmental practice, based on the sample analytical results. The laboratory will maintain proper records of waste disposal methods, and will have disposal company contracts on file for inspection.

## **9.0 DATA MANAGEMENT**

### **9.1 Data Management Applications**

All data generated as part of the Phase IV sampling will be maintained in an OU3-specific Microsoft® Access database. This will be a relational database with tables designed to store information on station location, sample collection details, preparation and analysis details, and analytical results. Results will include all asbestos data, including detailed structure attributes for TEM analyses.

As needed, EPA staff and designated contractors will develop tabular and graphical data summaries, perform statistical analyses, and generate maps using commercially-available applications such as Microsoft® Access and Excel and ArcGIS®.

### **9.2 Roles and Responsibilities for Data Flow**

#### **9.2.1 Field Personnel**

W.R. Grace Contractors will perform all Phase IV sample collection in accordance with the project-specific sampling plan and SOPs presented above. In the field, sample details will be documented on hard copy media-specific FSDS forms and in field log books (see Section 6.1.1). COC information will be documented on hard copy forms (see Section 6.1.4). FSDS and COC information will be manually entered into a field-specific<sup>2</sup> OU3 database using electronic data entry forms. Use of electronic data entry forms ensures the accuracy of data entry and helps maintain data integrity. For example, data entry forms utilize drop-down menus and check boxes whenever possible. These features allow the data entry personnel to select from a set of standard inputs, thereby preventing duplication and transcription errors and limiting the number of available selections (e.g., media types). In addition, entry into a database allows for the incorporation of data entry checks. For example, the database will allow a unique sample ID to only be entered once, thus ensuring that duplicate records cannot be created.

Entry of FSDS forms and COC information will be completed weekly, or more frequently as conditions permit. Copies of all FSDS forms, COC forms, and field log books will be scanned and posted in portable document format (PDF) to a project-specific file transfer protocol (FTP) site weekly. This FTP site will have controlled access (i.e., user name and password are required) to ensure data access is limited to appropriate project-related personnel. File names for scanned FSDS forms, COC forms, and field log books will include the sample date in the format YYYYMMDD to facilitate document organization (e.g., FSDS\_20090831.pdf).

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<sup>2</sup> The field-specific OU3 database is a simplified version of the master OU3 database. This simplified database includes only the station and sample recording and tracking tables, as well as the FSDS and COC data entry forms.

After FSDS data entry is completed, a copy of the field-specific OU3 database will be posted to the project-specific FTP weekly, or more frequently as conditions permit. The field-specific OU3 database posted to the FTP site will include the post date in the file name (e.g., FieldOU3DB\_20090831.mdb).

### 9.2.2 Laboratory Personnel

Each of the laboratories performing analyses for the Phase IV sampling are required to utilize all applicable Libby-specific Microsoft® Excel spreadsheets for data recording and electronic submittals (see Section 8.2.3). Upon completion of the appropriate analyses, EDDs will be transmitted via email to a designated email distribution list within the appropriate turn-around-time. Hard copies of all analytical laboratory data packages will be scanned to a PDF and either posted to the project-specific FTP site or emailed to a designated email distribution list. File names for scanned analytical laboratory data packages will include the laboratory name and the job number to facilitate document organization (e.g., LabX\_12365-A.pdf).

The email distribution list is as follows:

LibbyOU3@srcinc.com  
Lavelle.bonita@epa.gov  
Robert.r.marriam@grace.com

### 9.2.3 Database Administrators

Day-to-day operations of the master OU3 database will be under the control of EPA contractors. The primary database administrator will be responsible for sample tracking, uploading new data, performing error checks, and making any necessary data corrections. New records will be added to the master OU3 database within an appropriate time period of FSDS and/or EDD receipt.

Incremental backups of the master OU3 database will be performed daily Monday through Thursday, and a full backup will be performed each Friday. The full backup tapes will be stored off-site for 30 days. After 30 days, the tape will be placed back into the tape library to be overwritten by another full backup.

Each Friday, a copy of the master OU3 database will be posted to a project-specific FTP site to allow timely access to results by data users. The master OU3 database posted to the FTP site will include the post date in the file name (e.g., MasterOU3DB\_20090831.mdb).

## 9.3 **Data Storage**

All original data records (both hard copy and electronic) will be cataloged and stored in their original form until otherwise directed by the EPA Project Manager. At the termination of this

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project, all original data records will be provided to the EPA Project Manager in a format specified by EPA for incorporation into the site project files.

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## **10.0 ASSESSMENT AND OVERSIGHT**

Assessments and oversight reports to management are necessary to ensure that procedures are followed as required and that deviations from procedures are documented. These reports also serve to keep management current on field activities. Assessment, oversight reports, and response actions are discussed below.

### **10.1 Assessments**

#### **10.1.1 Field Oversight**

All individuals who collect samples during field activities will be provided a copy of this SAP and will be required to participate in a pre-sampling readiness review meeting to ensure that methods and procedures called for in this SAP and associated SOPs are understood and that all necessary equipment is on hand. EPA may perform random and unannounced field audits of field sampling collection activities, as may be deemed necessary.

#### **10.1.2 Laboratory Oversight**

All laboratories selected for analysis of samples for asbestos will be part of the Libby analytical team for OU3. These laboratories have all demonstrated experience and expertise in analysis of LA in environmental media, and all are part of an on-going site-specific quality assurance program designed to ensure accuracy and consistency between laboratories. These laboratories are audited by EPA and NVLAP on a regular basis. Additional laboratory audits may be conducted upon request from the EPA, as may be needed.

### **10.2 Response Actions**

If any inconsistencies or errors in field or laboratory methods and procedures are identified, response actions will be implemented on a case-by-case basis to correct quality problems. All response actions will be documented in a memo to the EPA RPM for OU3 at the following address:

Bonita Lavelle  
U.S. EPA, Region 8  
1595 Wynkoop Street  
Denver, CO 80202-1129  
E-mail: [lavelle.bonita@epa.gov](mailto:lavelle.bonita@epa.gov)

Any problems that cannot be corrected quickly through routine procedures may require implementation of a corrective action request (CAR) form.

### **10.3 Reports to Management**

Field and analytical staff will promptly communicate any difficulties or problems in implementation of the SAP to EPA, and may recommend changes as needed. If any revisions to this SAP are needed, the EPA RPM will approve these revisions before implementation by field or analytical staff.

## **11.0 DATA VALIDATION AND USABILITY**

### **11.1 Data Validation and Verification Requirements**

Data validation consists of examining the sample data package(s) against pre-determined standardized requirements. The validator may examine, as appropriate, the reported results, QC summaries, case narratives, COC information, raw data, initial and continuing instrument calibration, and other reported information to determine the accuracy and completeness of the data package. During this process, the validator will verify that the analytical methodologies were followed and QC requirements were met. The validator may recalculate selected analytical results to verify the accuracy of the reported information. Analytical results will then be qualified as necessary.

Data verification includes checking that results have been transferred correctly from laboratory data printouts to the laboratory report and to the EDD. Some of the data verification checks are performed as a function of built-in quality control checks in the Libby-specific data entry spreadsheets. Additional verifications of field and analytical results will be performed at a frequency of 10%. This initial rate may be revised as samples are analyzed and results evaluated. Data validation, review, and verifications must be performed on sample results before distribution to the public for review.

### **11.2 Reconciliation with Data Quality Objectives**

Once all samples have been collected and analytical data has been generated, data will be evaluated to determine if DQOs were achieved. Evaluation of the Phase IV data will include a qualitative and quantitative review of all QC samples and all deviations from sampling and analysis plans described in this report, along with conclusions regarding the reliability of the data for their intended use. Results of the data quality evaluation will in general be reported in the Baseline Human Health Risk Assessment, the Baseline Ecological Risk Assessment, and the final RI Report for OU3.



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### Panel A: Assumed Exposure Parameters

Parameter	Population									
	USFS Worker			USFS Firefighters				Rec Vis. to LRC	Residential wood harvesters	
	Trail maintenance	Tree thinning	Stand Examination	Cutting Fireline by Hand	Cutting Fireline w Heavy Equip.	Smoke on the ground	Smoke in air (Pilots)	Hiking along LRC	Driving to/from	Fell, cut, stack wood
ET (hrs/day)	8	8	8	8	8	8	4	8	1	8
EF (days/yr)	10	10	10	10	10	10	20	20	10	10
TWF	0.009	0.009	0.009	0.009	0.009	0.009	0.009	0.018	0.0011	0.009
Age at first exposure	18	18	18	18	18	18	18	7	15	15
Duration of exposure	40	40	40	40	40	40	40	30	30	30
iURad (PCM f/cc) <sup>-1</sup>	0.090	0.090	0.090	0.090	0.090	0.090	0.090	0.129	0.093	0.093

### Panel B: Risk Based Concentrations

Parameter	Population									
	USFS Worker			USFS Firefighters				Rec Vis. to LRC	Residential wood harvesters	
	Trail maintenance	Tree thinning	Stand Examination	Cutting Fireline by Hand	Cutting Fireline w Heavy Equip.	Smoke on the ground	Pilots	Hiking along LRC	Driving to/from	Fell, cut, stack wood
Target Risk	1.0E-05	1.0E-05	1.0E-05	1.0E-05	1.0E-05	1.0E-05	1.0E-05	1.0E-05	1.0E-05	1.0E-05
TWF	0.009	0.009	0.009	0.009	0.009	0.009	0.009	0.018	0.001	0.009
iURad	0.090	0.090	0.090	0.090	0.090	0.090	0.090	0.129	0.093	0.093
RBF	0.51	0.51	0.51	0.51	0.51	0.51	0.51	0.51	0.51	0.51
RBC (LA f/cc)	0.024	0.024	0.024	0.024	0.024	0.024	0.024	0.008	0.187	0.023

### Panel C: Target Sensitivity

Parameter	Population									
	USFS Worker			USFS Firefighters				Rec Vis. to LRC	Residential wood harvesters	
	Trail maintenance	Tree thinning	Stand Examination	Cutting Fireline by Hand	Cutting Fireline w Heavy Equip.	Smoke on the ground	Pilots	Hiking along LRC	Driving to/from	Fell, cut, stack wood
Target Fibers/sample	5	5	5	5	5	5	5	5	5	5
RBC (s/cc)	0.024	0.024	0.024	0.024	0.024	0.024	0.024	0.008	0.187	0.023
Target S (cc) <sup>-1</sup>	0.0048	0.0048	0.0048	0.0048	0.0048	0.0048	0.0048	0.0017	0.0373	0.0047

### Panel D: Estimated Grid Openings to Achieve Target Sensitivity

Parameter	Population									
	USFS Worker			USFS Firefighters				Rec Vis. to LRC	Residential wood harvesters	
	Trail maintenance	Tree thinning	Stand Examination	Cutting Fireline by Hand	Cutting Fireline w Heavy Equip.	Smoke on the ground	Pilots	Hiking along LRC	Driving to/from	Fell, cut, stack wood
EFA (mm2)	385	385	385	385	385	385	385	385	385	385
Ago (mm2)	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
Q (L/min)	4.0	4.0	4.0	4.0	4.0	4.0	4.0	4.0	4.0	4.0
time (min)	30	30	30	30	30	30	30	60	40	30
V (L)	120	120	120	120	120	120	120	240	160	120
Target S (cc)-1	0.0048	0.0048	0.0048	0.0048	0.0048	0.0048	0.0048	0.0017	0.0373	0.0047
GOs	68	68	68	68	68	68	68	97	7	69

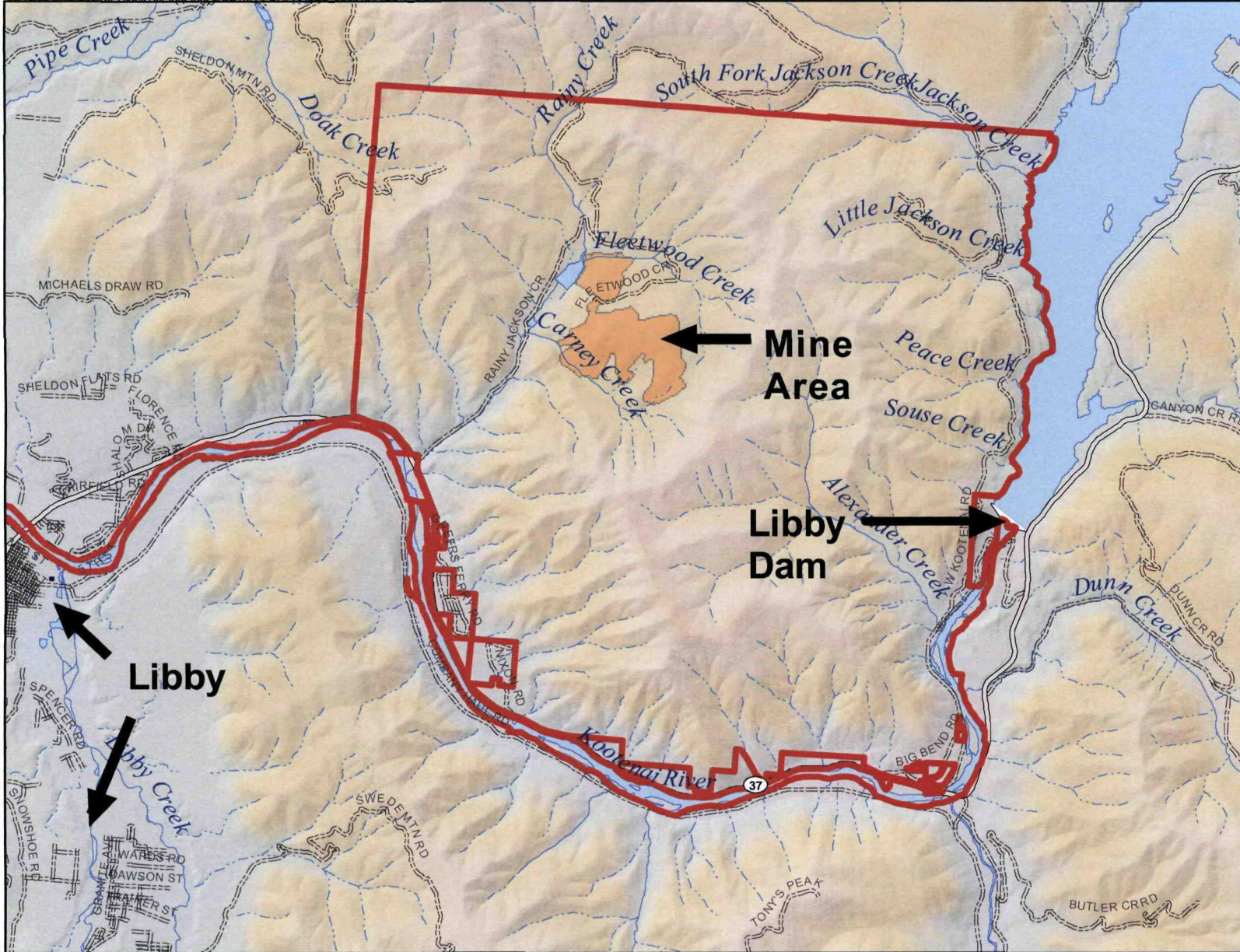
**TABLE 5-1 OVERVIEW OF PHASE IV ABS SAMPLING DESIGN**

Population	ABS Scenario	ABS Script	Number of ABS Study Areas	Number of ABS Events per Area	Number of samples (a)	Number of Samples	
						per area	total
Recreational visitor along Rainy Creek	Hiking along Rainy Creek	1	1	5	2	10	10
Residential wood harvester	Driving to and from harvest area	2a	3	5	2	10	30
	Cutting and hauling firewood	2b	3	5	4	20	60
USFS Worker (forest management activities)	Trail maintenance	3a	3	5	2	10	30
	Thinning trees	3b	3	5	2	10	30
	Stand exam	3c	3	5	2	10	30
USFS Firefighter (ground-based)	Cutting firelines by hand	3d	3	5	2	10	30
	Cutting firelines with heavy equipment	3e	3	5	2	10	30
	Personal monitors worn during simulated wildfire burns	4a	2	1	4	4	8
	Stationary monitors activated during simulated wildfire burns	4b	2	1	4	4	8
USFS Firefighter (pilot of aircraft)	Fly through smoke from simulated wildfires	5a	2	1	2	2	4
	Fly through smoke from authentic wildfires	5b	TBD	TBD	2	2	TBD
Area Residents and campers	Smoke monitoring near town from authentic wildfires	6	3	TBD	2	2	TBD
	Smoke monitoring downwind from authentic wildfires	6	TBD	1	1	1	TBD

(a) Note: Although each individual performing ABS activities will wear two pumps, it is assumed that only one of the resulting filters will be analyzed by TEM. The other filter will be archived for analysis if needed.

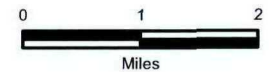
Planned	270
TBD (est)	30
Total (est)	300





### Legend

- Operable Unit 3
- County Road
- Primary Road
- Open Water
- Perennial Stream
- Intermittent Stream
- Mined Area



**Libby Montana Superfund Site**  
Operable Unit 3

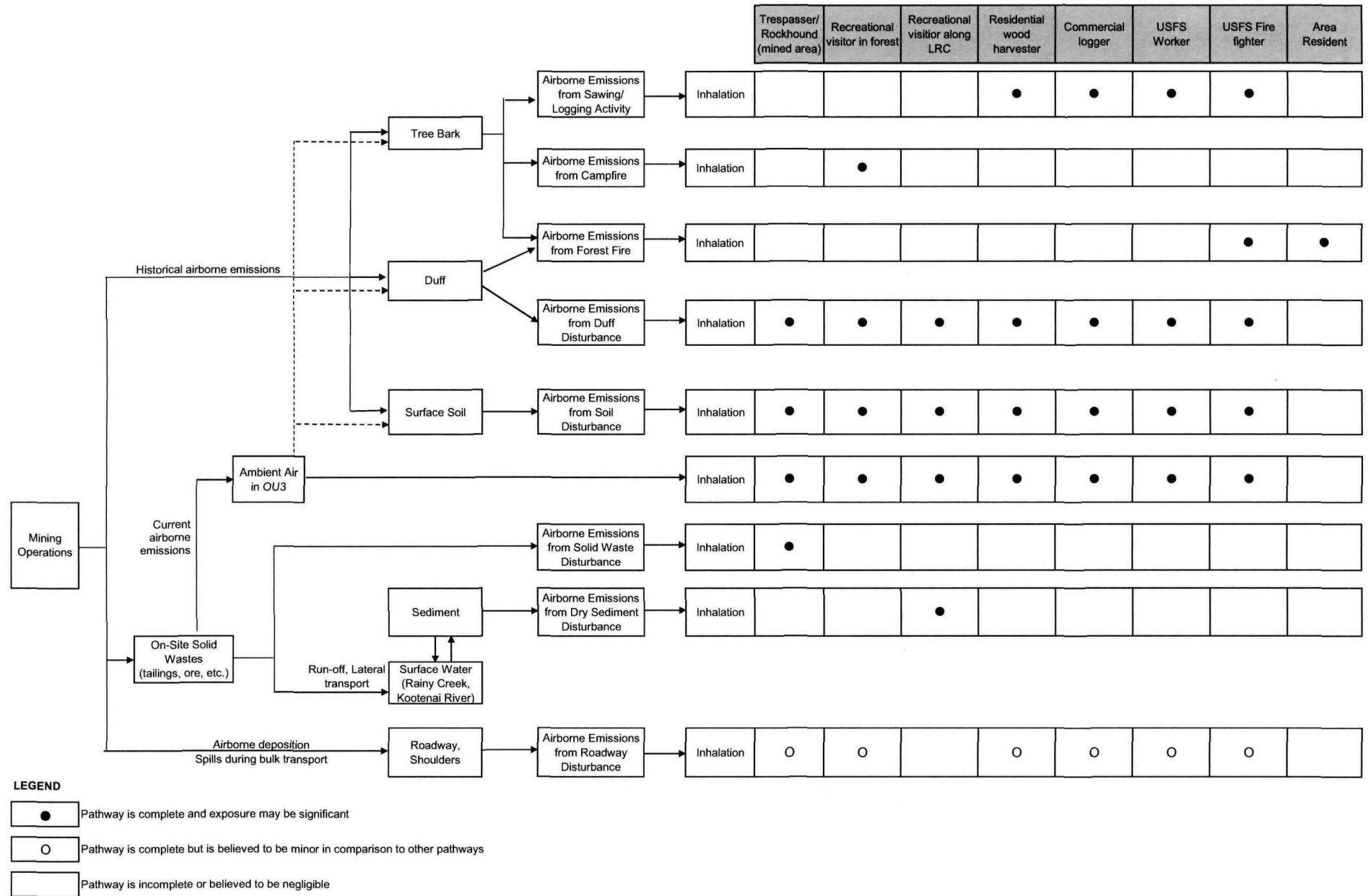
FIGURE 2-1

**Operable Unit 3**

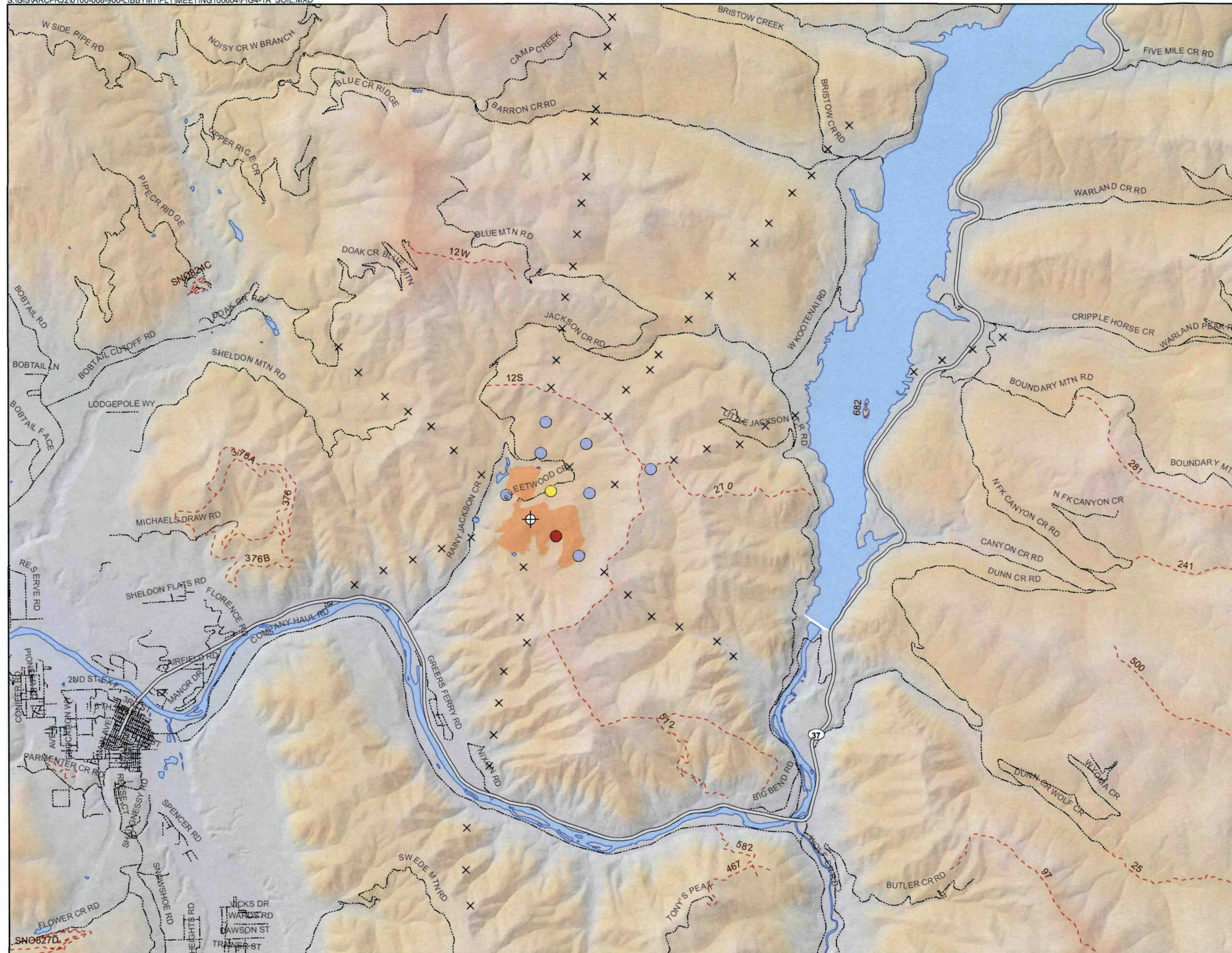
PROJECT: 0100-008-900	MAR 18, 2008
REV: 0	BY: VFS CHECKED: ACK



**Figure 3-1. Conceptual Site Model for Human Exposure to Asbestos**



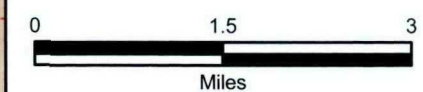




## Legend

**Asbestos in Forest Soil**  
(MFLA% fine - Percent Mass Fraction)

- × ND
- >0% and <=0.2%
- >0.2% and < 1%
- >= 1%



**LIBBY MONTANA SUPERFUND SITE**  
OPERABLE UNIT 3

FIGURE 4-1A

**ASBESTOS CONCENTRATIONS IN  
FOREST SOIL**

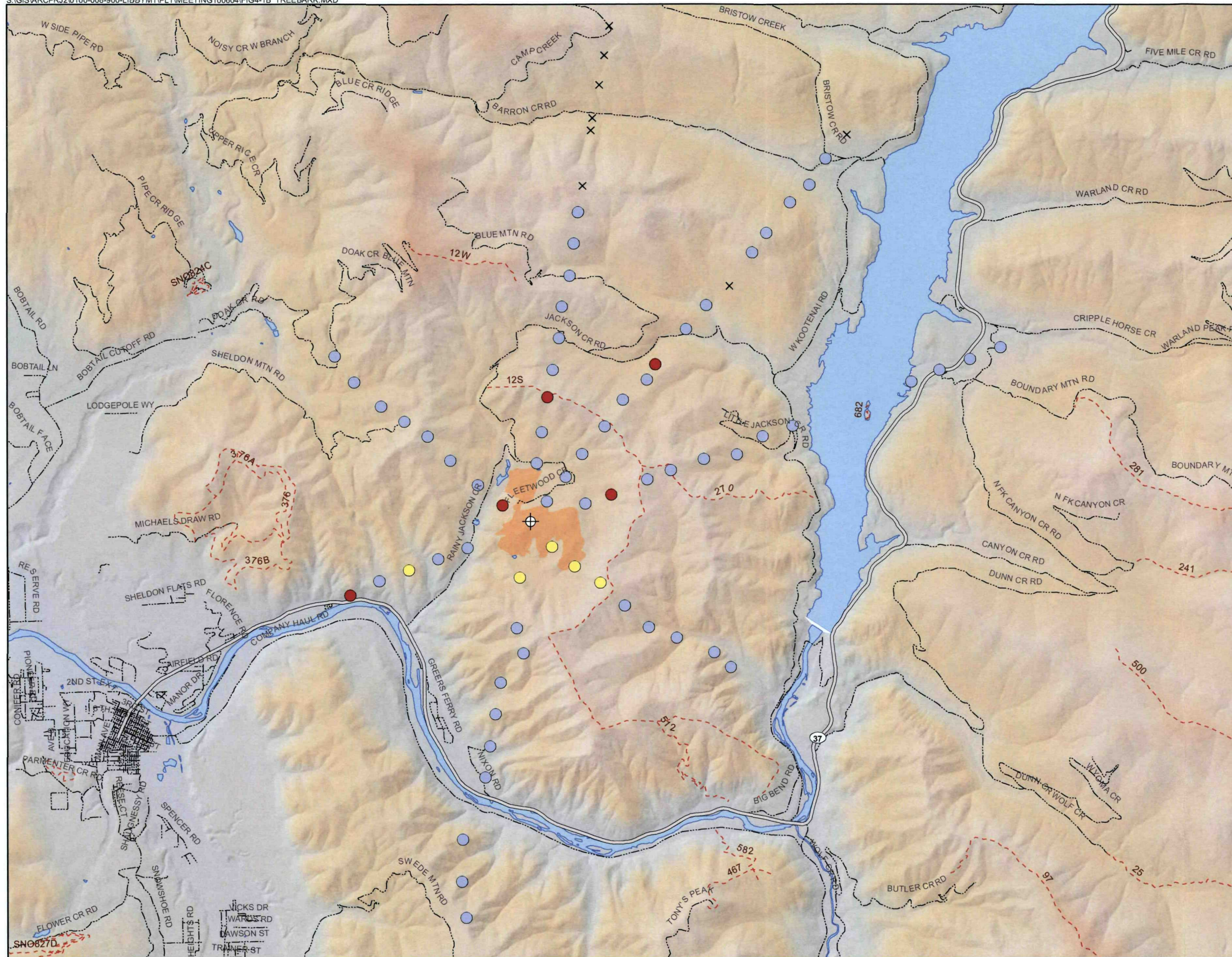
DATE: JUN 11, 2010

BY: CRL FOR: BL

**FORMATION**

ENVIRONMENTAL





**Legend**

**Asbestos in Tree Bark**  
(LA Loading - Million Structures/square cm)

- x 0.00
- 0.01 - 5.30
- 5.31 - 8.60
- 8.61 - 16.19

0 1.5 3  
Miles

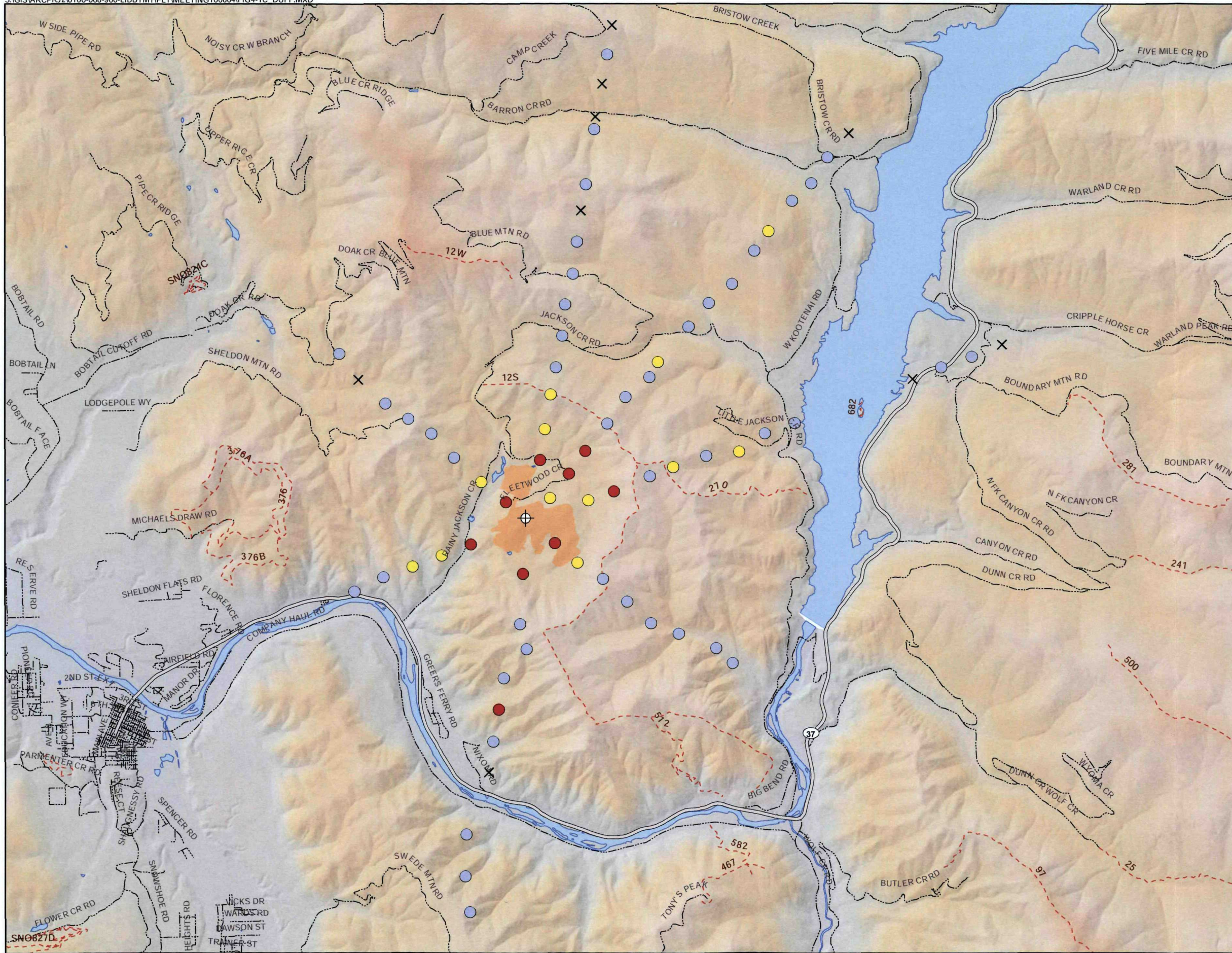
**LIBBY MONTANA SUPERFUND SITE**  
OPERABLE UNIT 3  
FIGURE 4-1B

**ASBESTOS CONCENTRATIONS IN TREE BARK**

DATE: JUN 11, 2010  
BY: CRL FOR: BL

**FORMATION**  
ENVIRONMENTAL





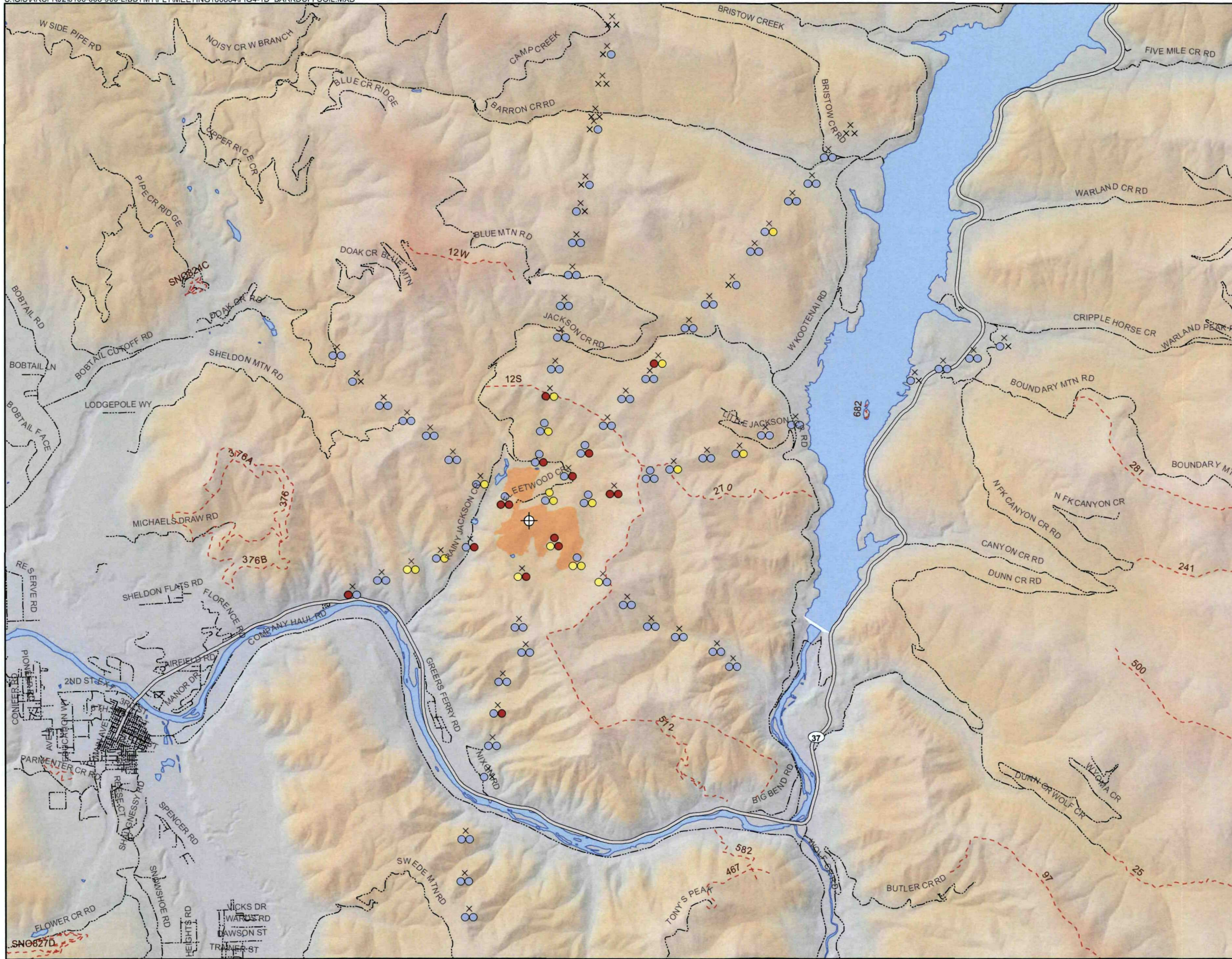
**Legend**  
**Asbestos in Duff**  
**(MFLA% Dried - % Mass Fraction)**  

- X ND
- >0% and <= .2%
- >= .2% and <1%
- >= 1%

**LIBBY MONTANA SUPERFUND SITE**  
**OPERABLE UNIT 3**  
FIGURE 4-1C  
**ASBESTOS CONCENTRATIONS IN DUFF**

DATE: JUN 11, 2010	<b>FORMATION</b> ENVIRONMENTAL
BY: CRL FOR: BL	





**Legend**

**Asbestos in Forest Soil**  
(MFLA% fine - % Mass Fraction)

- × ND
- >0% and <=0.2%
- >0.2% and < 1%
- >= 1%

**Asbestos in Tree Bark**  
(LA Loading - Million Structures/square cm)

- × 0.00
- 0.01 - 5.30
- 5.31 - 8.60
- 8.61 - 16.19

**Asbestos in Duff**  
(MFLA% Dried - % Mass Fraction)

- × ND
- >0% and <= .2%
- >= .2% and <1%
- >= 1%

**Symbol Placement**

- Forest Soil
- Tree Bark
- Duff

0 1.5 3  
Miles

**LIBBY MONTANA SUPERFUND SITE**  
OPERABLE UNIT 3  
FIGURE 4-1D  
**ASBESTOS CONCENTRATIONS IN  
FOREST SOIL, TREE BARK,  
AND DUFF**

DATE: JUN 11, 2010  
BY: CRL FOR: BL

**FORMATION**  
ENVIRONMENTAL



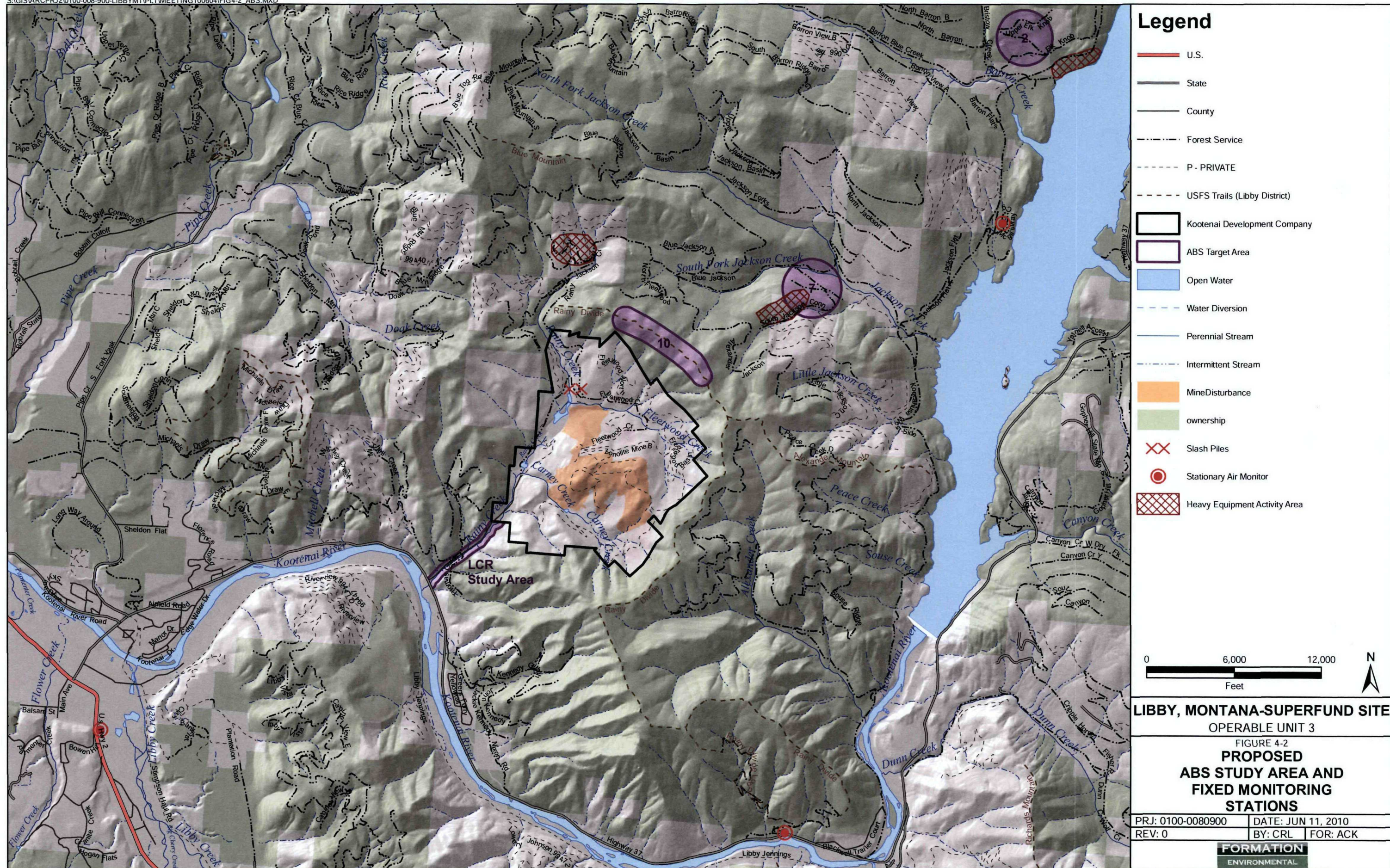




FIGURE 4-3  
SLASH PILE 1 IN OU3



FIGURE 4-4  
SLASH PILE 2 IN OU3





**LIBBY SUPERFUND SITE OPERABLE UNIT 3  
PHASE IV SAMPLING AND ANALYSIS PLAN**

**ATTACHMENT A**

**ACTIVITY BASED SAMPLING (ABS) SCRIPTS**

This Attachment describes the activities to be performed by individuals performing each of the ABS scenarios described in the Phase IV-A SAP. Details on the number, location, and timing of ABS sampling events are provided in the Phase IV-A SAP.

**SCRIPT 1: RECREATIONAL VISITORS ALONG LOWER RAINY CREEK**

Recreational visitors along lower Rainy Creek (between Highway 37 and the W.R. Grace property line) may engage in activities such as hiking and antler hunting that could disturb LA in source material (soil, dried stream-side sediment, and duff). The following script will be used to simulate exposures during activities that are considered to be representative of recreational visitors along lower Rainy Creek south of the W.R. Grace property boundary.

For this scenario, Rainy Creek south of the W.R. Grace property boundary will be evaluated as a single ABS study area. ABS sampling will be performed by a team of two individual samplers. Two pumps will be worn by each sampler. Target flow rate for the first pump is 2 liters per minute (LPM) and the target flow rate for the second pump is 4 LPM.

The team of two samplers will walk from the security gate on Rainy Creek Road to Rainy Creek. Once at Rainy Creek, the two samplers will turn on their sampling pumps. This will be time = 0. Both individuals will then begin to walk up along the banks of the creek, disturbing bushes and other vegetation as needed to move along the bank of the creek. Activities will include the samplers using their hands to push aside ground vegetation to simulate hunting for antlers. To the extent possible, the samplers will cross back and forth across the creek several times so that the ABS activities represent exposures to disturbed sediments and brush on both sides of Rainy Creek.

After 30 minutes moving up the drainage, the two samplers will change positions so that the sampler originally in the lead will be in the following position. The team will then make their way back down the drainage toward the starting point. After 30 more minutes (for a total of 60 minutes), the ABS event ends and the air sampling pumps are turned off and the air sampling cassettes are capped.

## SCRIPT 2: RESIDENTIAL WOOD HARVESTER

Residents of Libby may harvest firewood within the OU3 study area for personal use as a heating source. Individuals harvesting wood within the OU3 study area may be exposed to LA fibers released from duff and/or soil when disturbed during walking in the woods to the selected trees, from tree bark when cutting down trees and sawing them into manageable size, and from tree bark and/or soil and/or duff when disturbed during hauling the wood to a truck or other vehicle. The following script will be used to simulate residential (non-commercial) wood harvesting activities.

Two individual samplers will perform each residential wood harvesting ABS event. Two pumps will be worn by each sampler. The target flow rate for the first pump is 2 LPM and the target flow rate for the second pump is 4 LPM.

For each ABS event at each location, each individual sampler will collect 1 sample to represent exposures that occur while driving to and from the location, and 2 additional samples during the harvesting (felling the tree, cutting it into manageable firewood sizes, stacking into the back of a pickup truck, and cleaning up the area). The scenario is performed in three steps, as follows:

### *1. Driving to the ABS Area*

The individual samplers shall initiate the event by entering a pickup truck located in the Flyway, turning on their air monitoring pumps, and riding to the location of trees to be harvested (identified by the US Forest Service ) in the truck with the windows open. Once arriving at the tree harvesting location, the truck will be parked along the US Forest Service road, the air monitoring pumps will be turned off and the air sampling cassettes will be capped and set aside until they are re-activated during the drive away from the ABS area.

### *2. Felling, Cutting and Stacking*

The wood harvesting scenario is begun by each individual exiting the pickup truck, attaching new filter cassettes to the low volume and the high volume pumps, and turning the pumps on.

During each ABS event at each designated location, the samplers will harvest one tree identified by the US Forest Service. The samplers shall hike to the tree and one of the samplers will fell the tree and remove branches using a chainsaw. The second individual will haul the cut branches and place them in a pile nearby. The duration of this activity will vary depending on the size of the tree and the distance from the road, but is expected to average approximately 20 minutes. After felling the tree and removing the branches, the samplers shall attach the cut tree to a cable and haul it to the road near the vehicle. Then one sampler will cut the felled tree into pieces of appropriate size for use in wood burning stoves (usually about 16-24 inches in length) and the other sampler will haul the

firewood back to the bed of the truck. After 15 minutes, the samplers will reverse roles and continue for another 15 minutes. After this time, all smaller branches and other small debris from the tree will be scattered at random off the road. During the cleanup, samplers shall collect samples for a period of 10 minutes. The total duration for this activity is 60 minutes.

In order to help minimize the chances of generating overloaded filters, the exposures associated with the wood harvesting activities described above will be captured on two sequential cassettes. The optimal break point between the two sequential cassettes will be determined in a pilot study to be conducted before authentic ABS sampling to support the remedial investigation are collected. For example, the first wood harvesting sample may include hiking to the tree, felling, and hauling to the road, and the second wood harvesting sample may include cutting, stacking, and clean up. Other combinations are acceptable as long as all activities are sampled.

All individuals who perform this activity must be properly trained in the safe use of gasoline powered saws and in safe procedures for felling trees.

After 60 minutes of cutting and hauling wood to the truck and cleaning up, the harvesting scenario is ended. Each individual shall turn off their pumps and remove and cap the cassettes. The area shall be further cleaned up as necessary after sampling has ended.

### *3. Driving from the ABS Area*

After removing the cassettes used for wood harvesting, each individual shall re-attach the same cassettes as were used during the truck ride to the area. The pumps will then be re-activated, and the pickup truck shall be driven back to the Flyway (windows open). Once at the Flyway, the driving scenario is ended and the pumps will be turned off and each cassette removed and capped.

At the Flyway, the wood shall be off-loaded from the truck and placed into a pile for potential future use in burning<sup>1</sup>. The wind direction and speed at the sampling location should also be monitored.

## **SCRIPT 3: US FOREST SERVICE WORKER**

U.S. Forest Service workers within the OU3 study area may be exposed to LA fibers during a variety of activities. These include activities routinely performed as part of the Forest Service's land management responsibilities such as maintenance of roads and trails, thinning of trees and vegetation, and surveying trees (stand exam). In addition to routine land management activities,

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<sup>1</sup> The potential risks associated with burning wood collected from OU3 will be evaluated as part of the OU4 risk assessment, and will not be included in OU3.

US Forest Service personnel respond to forest fires in the Kootenai National Forest. Fire fighting activities include those associated with initial ground attack, aerial attack, and sustained ground attack.

For the purposes of the Phase IV ABS investigation, the following scripts will be used to simulate a range of activities that are considered to be representative. ABS sampling will be performed by a team of two samplers except where noted. Two pumps will be worn by each sampler. The target flow rate for the first pump is 2 LPM and the target flow rate for the second pump is 4 LPM.

#### Script 3a. Trail Maintenance

Trail maintenance consists mainly of clearing vegetation from the trail. Samplers will travel to the designated ABS locations and park on the roadway. Samplers will exit the vehicle and hike to the designated trail location. Once at the designated trail, samplers will turn on the air sampling pumps. One sampler will use a small chain saw and the other sampler will use a saw head trimmer to clear trees and brush to a width of about 1-2 feet, and to a height of about 8 feet. The second individual will haul the cut vegetation off the trail. After 15 minutes, the two individuals shall reverse roles. After 30 minutes, the ABS activity is ended, and both individuals shall turn off their air sampling pumps and cap their sampling cassettes.

#### Script 3b. Tree Thinning

Tree thinning consists of cutting small trees (2-4 inches in diameter) and hauling the wood into a "slash" pile. This activity will be performed for a period of 30 minutes. One individual will cut for 15 minutes, while the second individual hauls wood to a pile. After 15 minutes, the roles are reversed. After 30 minutes, the ABS activity is ended, and both individuals shall turn off their air sampling pumps and cap their sampling cassettes.

#### Script 3c. Stand Examination

Stand examination consists of obtaining measurements of the girth and height of trees in a stand, and collecting borings from the trees to determine age. Both samplers will perform all of these activities. Tree diameter is measured by passing a measuring tape around the tree at a height of about 1 meter. Tree height is derived by measuring to a reference point about 10-15 meters from the tree, and measuring the angle from that point to the top of the tree with a clinometer. Collection of a core using an increment borer is described in SOP Tree-Libby-OU3 (see Attachment B). This scenario will be performed by both samplers for a period of 30 minutes. After 30 minutes, the ABS activity is ended, and both individuals shall turn off their air sampling pumps and cap their sampling cassettes.

#### Script 3d. Cutting a Fireline by Hand

Cutting a fireline by hand consists of establishing a fuel break 18 inches wide by removing as much combustible material as possible. This task requires initial removal of trees and brush using a chainsaw. Then a Pulaski tool or other similar device is used to scrape away all combustible material down to mineral soil to establish a line about approximately 18 inches wide. During an initial attack of a forest fire, these activities are typically done by a crew of 4-6 fire fighters. For the ABS scenario, 2 samplers will participate. This activity will be performed for a period of 30 minutes. The 2 samplers will work approximately 10 feet apart. After 15 minutes, the relative positions of the 2 samplers will be reversed. After 30 minutes, the ABS activity is ended, and both samplers shall turn off their air sampling pumps and cap their sampling cassettes.

#### Script 3e. Cutting a Fireline with Heavy Equipment

Cutting a fireline with heavy equipment involves using a dozer or tractor plow to remove vegetation and all combustible material down to mineral soil within a line as wide as the dozer or tractor blade. This activity is typically done by a crew consisting of one dozer boss and a crew of several fire fighters. For the ABS scenario, 2 samplers will participate. One sampler will act as the dozer boss and will operate the dozer or tractor plow to cut the fireline. The other sampler will perform activities to simulate a firefighter. The second sampler shall be no closer than 100 feet from the heavy equipment and will pick up and discard excavated combustible material from inside the fireline to outside the fireline. This activity will be performed for a period of 30 minutes. After 30 minutes, both samplers shall turn off their air sampling pumps and cap their sampling cassettes. The dozer/tractor plow operator will then pull back the excavated lines to clean up the area.

#### **SCRIPT 4: EXPOSURE OF GROUND-BASED FIREFIGHTERS TO SMOKE**

U.S. Forest Service workers who fight fires on the ground within the OU3 study area may be exposed to LA in air released by burning of contaminated duff and trees. This "smoke" exposure pathway will be evaluated after the results from Scripts 3d and 3e are available and have been evaluated.

The objective of this activity is to characterize LA levels in smoke released from high-intensity fires. This will be achieved by burning two large slash piles that presently exist in OU3. All such simulated wildfire burns in OU3 will be performed in accord with all requirements and recommendations of the U.S. Forest Service. One or more U.S. Forest Service staff will be present at all simulated wildfire events.

#### Script 4a. Personal ABS Samples

Four samplers will wear personal air monitors during each simulated wildfire. Two pumps will be worn by each sampler. The target flow rate for the first pump is 2 LPM and the target flow rate for the second pump is 4 LPM. The pumps will not be activated until the fire is generating significant levels of smoke. When smoke generation is significant, the pumps will be activated and each individual wearing the monitors will move about the area of the controlled burn, including time in the cross-wind and down-wind directions. The primary purpose is to capture exposures related to smoke release rather than soil or duff disturbance. After 30 minutes, the ABS activity is ended, and both samplers shall turn off their air sampling pumps and cap their sampling cassettes.

#### Script 4b. Stationary Monitors

Four stationary monitors will be installed around the perimeter of each simulated wildfire. These will be positioned approximately 90° apart, with one station being located in the predominant downwind direction at the time the fire is initiated. Each perimeter monitor should be sufficiently distant from the fire that it is not threatened by the heat from the fire. The perimeter monitors will not be activated until the fire is generating significant levels of smoke. Once the pumps are activated, each sample will be collected for a period of 30 minutes. After 30 minutes, the stationary pumps will be turned off and the sampling cassettes capped.

In addition, if smoke from either of the simulated wildfires is blowing towards any of the three fixed-base contingency monitors described in Attachment D (See Section 4.1 of Attachment D), one or more of these monitors should also be activated during the simulated wildfire.

Following the fire, stationary air samples will be collected from each of the four perimeter air monitors for 2 additional days to determine if fibers remain in the air or are dispersed by the wind. Because smoke emission will be much lower than during the fire, sampling time can be increased from 30 minutes to 60 minutes per sample to help decrease the analytical sensitivity.

### **SCRIPT 5: EXPOSURE OF PILOTS TO SMOKE**

Currently, within the Fire Suppression Restricted Zone (FSRZ) of the OU3 study area, U.S. Forest Service personnel fight forest fires by air, either in fixed wing aircraft or in helicopters that are used to drop water or fire retardant on burning areas. These individuals may be exposed to LA that has been released into the air by the fire.

In order to characterize the exposure of aircraft pilots during fire suppression flights, a stationary sampling pump will be installed in the cockpit of one or more aircraft deployed for fighting fires within the OU3 study area. The air sampling cassette will be positioned to sample cockpit air,

but will be located in a position that does not interfere with the pilot's vision or ability to operate the aircraft.

#### Script 5a. Responding to Simulated Wildfires in OU3

On the day(s) when simulated wildfires are performed in OU3 (see Script 4), one aircraft shall fly over the simulated wildfire area after the smoke plume is well established, simulating a flight path that would be used to attack the fire. Two such simulated aerial attacks shall be performed per simulated wildfire, resulting in 2 filters per wildfire (four total).

For each simulated attack, the air sampling pump will be turned on when the pilot is preparing for takeoff, and will be turned off when the pilot returns to base. For each sample collected, the time of the flight (from takeoff to landing) will be recorded, along with information on the location of the fire and the type of aircraft.

#### Script 5b. Responding to Authentic Wildfires in OU3

When an aerial response to an authentic wildfire in the exclusion zone of OU3 is called for, the USFS will notify Remedium. If the fire is generating significant smoke, Remedium will immediately send a person to the airfield to perform all necessary activities associated with calibrating and activating the pump and collecting the samples (as described above). It is understood that, in some cases, the pilot may be required to begin flights before this can be achieved. In this event, the pump will be activated during the first available time when the aircraft returns to base between trips to the fire.

### **SCRIPT 6: EXPOSURE OF AREA RESIDENTS TO SMOKE FROM FOREST FIRES**

EPA (2009b) previously established a plan for collecting stationary air samples at three monitoring locations to evaluate exposures of residents in Libby in the event of a significant forest fire in the Fire Suppression Restricted Zone of OU3. This original "contingency air monitoring plan" has been revised to incorporate the addition of one mobile air monitoring station that will be placed in a downwind location from any authentic wildfire that generates significant smoke in OU3. This revised "contingency air monitoring plan" is provided as Attachment D to this SAP.

In brief, when notified by the USFS of a fire in the exclusion zone of OU3 that is large enough to generate significant smoke, a designated point of contact who is a contractor to Remedium will activate stationary air sampling pumps previously deployed at three locations:

- CDM office building in Libby
- USFS Ranger station along Highway 37
- McGillivray Campground on the west shore of Lake Koocanusa.

Unless smoke levels are so high as to cause filter overloading, sampling duration for these samples will be 24 hours, changing to new filters every 24 hours until the fire is extinguished.

In addition to these three stationary monitors at fixed locations, a fourth monitoring station capable of collecting two independent samples (field duplicates) will be established in an accessible area downwind of the fire. The monitors will be transported to the collection site by truck. The sampling location and distance from the fire will depend on the conditions of the fire. Although details may vary, it is envisioned that the two monitors will be placed on a tripod in the back of the truck, which will be driven to the nearest accessible area that is being impacted by smoke from the fire. Once at the sampling location, the pumps shall be calibrated and the pumps turned on. The target flow rate for these samplers will be 4-6 LPM. Unless safety concerns dictate otherwise, this sampler shall be collected for duration of about 30 minutes. During sample collection, the coordinates of the monitor will be recorded. This information will be used later, in combination with data on the fire location, to establish the distance and direction of the monitor relative to the fire. The wind direction and speed at the sampling location should also be monitored.

**NOTE: In all cases, it is critical that this sampling effort be performed in a way that does not endanger that health or safety of the sampling personnel. If conditions are considered to be potentially unsafe, the sampler should evacuate the area immediately.**



Libby Superfund Site Operable Unit 3 Standard Operating Procedure

Date: September 26, 2007

OU3 SOP 7 (Rev. 0)

Title: EQUIPMENT DECONTAMINATION

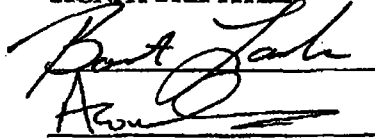
APPROVALS:

TEAM MEMBER

SIGNATURE/TITLE

DATE

EPA Remedial Project Manager



9/26/07

SOP Author



9/26/07

Revision Number	Date	Reason for Revision
0	09/26/2007	-

## 1.0 INTRODUCTION

This Standard Operating Procedure (SOP) is based on MWH SOP-02, Equipment Decontamination, Revision 1.0, March 2004, modified for use at the Libby Asbestos Superfund Site OU3. Decontamination of drilling, sampling, and monitoring equipment is a necessary and critical aspect of environmental field investigations. Proper decontamination is a key element in reducing the potential for cross-contamination between samples from different locations, as well as ensuring that samples are representative of the sampled materials. Improper decontamination may result in costly re-collection and re-analysis of samples. All equipment used in the sampling process will be properly decontaminated prior to the collection of each sample and after completion of sampling activities.

The procedures outlined in this SOP will be followed during decontamination of field equipment used in the sampling process, including drilling, soil/water sample collection, and monitoring activities. Any deviations from these procedures will be noted in the field notebooks and approved by the appropriate oversight agency, if significant. Three major categories of field equipment, along with applicable decontamination methods for each, are discussed below.

## 2.0 HEALTH AND SAFETY WARNING

All personnel engaged in equipment decontamination must follow health and safety protocols described in the health and safety plan. Asbestos fibers are thin and long fibers so small that they cannot be seen by the naked eye. Asbestos fibers are easily inhaled when disturbed and when embedded in the lung tissue can cause health problems. Significant exposure to asbestos increases the risk of lung cancer, mesothelioma, asbestosis (non-cancerous lung disease), and other respiratory diseases (ATSDR 2006).

## 3.0 DEFINITIONS

**Bailer:** A cylindrical tool designed to remove material from a well. A valve at the bottom of the bailer retains the contents in the bailer.

**Bladder Pump:** Groundwater sampling equipment consisting of a flexible bladder, usually made of Teflon<sup>®</sup>, contained within a rigid cylindrical body (commonly made of stainless steel). The lower end of the bladder is connected to the intake port through a check valve, while the upper end is connected through a second check valve to a sampling line that leads to the ground surface.

**Brass Sleeve:** Hollow, cylindrical sleeves made of brass and used as liners in split-spoon samplers for collection of undisturbed samples.

**Auger Flight:** An individual auger section, usually 5 feet in length.

**Continuous Core Barrel:** 3-5 foot long steel barrels that can be joined together to allow continuous cores to be collected during a single run.

**Drill Pipe:** Hollow metal pipe used for drilling, through which soil and groundwater sampling devices can be advanced for sample collection.

**Peristaltic Pump:** A low-volume suction pump. The compression of a flexible tube by a rotor results in the development of suction.

**Source Water:** A drilling quality water source identified to be used for steam cleaning. This source should be sampled at the beginning of each field program to set baseline concentrations.

**Distilled Water:** Commercially available water that has been distilled. Each batch of distilled water should be analyzed to set baseline concentrations.

**Hand Auger:** A sampling tool consisting of a metal tube with two sharpened spiral wings at the tip.

**Split-Spoon Sampler:** A sampling tool consisting of a thick-walled steel tube with a removable head and drive shoe. The steel tube splits open lengthwise when the head and drive shoe are removed.

**Scoop:** A sampling hand tool consisting of a small shovel- or trowel-shaped blade.

**Submersible Pump:** Groundwater sampling pump that consists of a rotor contained within a chamber and driven by an electric motor.

#### **4.0 RESPONSIBILITIES**

This section presents a brief definition of field roles, and the responsibilities generally associated with them. This list is not intended to be comprehensive and often, additional personnel may be involved. Project team member information will be included in project-specific plans (e.g., work plan, field sampling plan, quality assurance plan, etc.), and field personnel will always consult the appropriate documents to determine project-specific roles and responsibilities. In addition, one person may serve in more than one role on any given project.

**Project Manager:** Responsible for project implementation and coordination, selects project-specific drilling and sampling methods, and associated decontamination procedures with input from other key project staff, and appropriate oversight agencies.

**Quality Control Manager:** Overall management and responsibility for quality assurance and quality control (QA/QC). Selects QA/QC procedures for the sampling and analytical methods, performs project audits, and ensures that data quality objectives are fulfilled.

**Field Team Leader (FTL) and/or Geologist, Hydrogeologist, or Engineer:** Implements the field program and supervises other sampling personnel, and ensures that SOPs are properly followed. Prepares daily logs of field activities.

**Field Sampling Technician (or other designated personnel):** Assists the FTL, geologist, hydrogeologist, or engineer in the implementation of tasks and is responsible for the decontamination of sampling equipment.

## **5.0 DECONTAMINATION PROCEDURES**

Drilling and sampling procedures require that decontaminated tools be employed in order to prevent cross-contamination. The decontamination procedures described below will be followed to ensure that only uncontaminated materials will be introduced to the subsurface during drilling and sampling. The equipment decontamination process will be undertaken before and after each use of the equipment and include either steam cleaning or washing. Steam cleaning of equipment, if used, will be performed at a temporary decontamination site. The flooring of the temporary decontamination site will be impermeable to water and large enough to contain the equipment and the rinsate produced.

If the quantity of water in the pad area exceeds its holding capacity, the water will be drummed temporarily until analytical results are obtained and the water can be properly disposed of. Steam cleaning will not be performed over bare ground, but will always be conducted so that rinsate can be collected and disposed of properly. Wherever applicable, equipment will be disassembled to permit adequate cleaning of the internal portions.

### **5.1 Drilling and Large Equipment**

The following procedure will be used for decontamination of large pieces of equipment. These include well casings, auger flights, drill pipes and rods, and those portions of the drill rig that may stand directly over a boring or well location, or that may come into contact with casing, auger flights, pipes, or rods.

- Establish a decontamination area large enough to contain the equipment and any decontamination waste
- Place equipment on sawhorse or equivalent, if possible.

- Steam clean the external surfaces and internal surfaces, as applicable, on equipment using high-pressure steam cleaner from an approved water source. If necessary, scrub using brushes and a phosphate-free detergent (e.g., Alconox<sup>TM</sup>), or equivalent laboratory-grade detergent until all visible dirt, grime, grease, oil, loose paint, rust, etc., have been removed.
- Rinse with potable water
- Remove equipment from decontamination pad and allow to air dry
- Record date and time of equipment decontamination

## 5.2 Soil and Groundwater Sampling Equipment

The following procedure will be used to decontaminate sampling equipment such as split-spoon samplers; brass sleeves; continuous core barrels; scoops; hand augers; non-dedicated bailers; submersible pumps, bladder pumps; and other sampling equipment that may come into contact with samples. To minimize decontamination procedures in the field, dedicated equipment will be used wherever feasible:

- Wash and scrub equipment with phosphate-free, laboratory-grade detergent (e.g., Alconox<sup>TM</sup> or equivalent) and off-site distilled water
- Triple-rinse with distilled water
- Air dry
- Wrap in aluminum foil, or store in clean plastic bag or designated casing.

- Record date and time of equipment decontamination

Personnel involved in decontamination activities will wear appropriate protective clothing as defined in the project-specific health and safety plan.

### 5.3 Monitoring Equipment

The following procedure will be used to decontaminate monitoring devices such as slug-test equipment, groundwater elevation and free product thickness measuring devices, and water quality checking instruments. Note that organic solvents can not be used to decontaminate free product measuring devices because they will cause damage to the probes. Spray bottles may be used to store and dispense distilled water.

- Wash equipment with laboratory-grade, phosphate-free detergent (e.g., Alconox™ or equivalent) and distilled water
- Triple-rinse with distilled water
- Store in clean plastic bag or storage case.
- Record date and time of equipment decontamination

## 6.0 QUALITY ASSURANCE AND QUALITY CONTROL

All equipment decontamination must be documented in the field logbooks and/or field forms, including rationales deviations from this SOP. The Field Team Leader or designated QA reviewer will check and verify that field documentation has been completed per this procedure and other procedures referenced herein.

To assess the adequacy of decontamination procedures, field rinsate blanks may be collected. The specific number of rinsate blanks will be defined in a FSP or work plan or by the Project

Manager. In general, at least one field rinsate blank should be collected per sampling event or per day.

Rinsate blanks with elevated or detected contaminants will be evaluated by the Project Manager, who will relay the results to the site workers. Such results may be indicative of inadequate decontamination procedures that require corrective actions (e.g., retaining).

## 7.0 PROCEDURE FOR WASTE DISPOSAL

All decontamination water that has come into contact with contaminated equipment will be handled, labeled, stored and disposed according to SOP 12. Unless otherwise specified in the FSP, waste generated from other sources and classified as non-hazardous waste (e.g., PPE, plastic sheeting, rope and misc. debris) will be disposed into trash receptacles.

## 8.0 REFERENCES

Agency for Toxic Substances and Disease Registry. 2006. *Asbestos Exposure and Your Health*.

U.S. Environmental Protection Agency, *RCRA Ground-Water Monitoring: Draft Technical Guidance*, November 1992. Page 7-17.



**Libby Superfund Site Operable Unit 3 Standard Operating Procedure**

Date: September 26, 2007

OU3 SOP 8 (Rev. 0)

Title: SAMPLE HANDLING AND SHIPPING

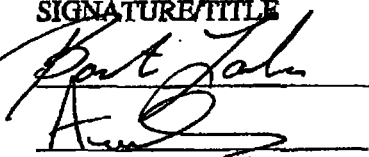
**APPROVALS:**

TEAM MEMBER

SIGNATURE/TITLE

DATE

EPA Remedial Project Manager



9/26/07

SOP Author

9/26/07

Revision Number	Date	Reason for Revision
0	09/26/2007	—

## 1.0 INTRODUCTION

This standard operating procedure (SOP) is based on MWH SOP-09, Sample Handling and Shipping, Revision 1.0, March 2004, modified for use at the Libby Asbestos Superfund Site OU3. This SOP describes the requirements for sample handling, storage and shipping. The purpose of this SOP is to define sample management activities as performed from the time of sample collection to the time they are received by the laboratory.

## 2.0 HEALTH AND SAFETY WARNING

All personnel engaged in soil sampling must follow health and safety protocols described in the health and safety plan. Asbestos fibers are thin and long fibers so small that they cannot be seen by the naked eye. Asbestos fibers are easily inhaled when disturbed and when embedded in the lung tissue can cause health problems. Significant exposure to asbestos increases the risk of lung cancer, mesothelioma, asbestosis (non-cancerous lung disease), and other respiratory diseases (ATSDR 2006).

## 3.0 DEFINITIONS

**Chain-of-Custody:** An accurate written record of the possession of each sample from the time of collection in the field to the time the sample is received by the designated analytical laboratory.

**Sample:** Physical evidence collected for environmental measuring and monitoring. For the purposes of this SOP, sample is restricted to solid, aqueous, air, or waste matrices. This SOP does not cover samples collected for lithologic description nor does it include remote sensing imagery or photographs (refer to SOP-9 for field documentation procedures).

**Sampler:** The individual who collects environmental samples during fieldwork.

## 4.0 RESPONSIBILITIES

This section presents a brief definition of field roles, and the responsibilities generally associated with them. This list is not intended to be comprehensive and often additional personnel may be involved. Project team member information will be included in project-specific plans (e.g., work plan, field sampling plan (FSP), quality assurance plan, and etc.), and field personnel will always consult the appropriate documents to determine project-specific roles and responsibilities. In addition, one person may serve in more than one role on any given project.

**Project Manager:** The Project Manager is responsible for ensuring that the requirements for sample management are included in the appropriate project plans. The Project Manager is responsible for coordinating sample management efforts with input from other key project staff and applicable government agencies.

**Quality Control Manager:** Overall management and responsibility for quality assurance and quality control (QA/QC). Selects QA/QC procedures for the sampling and analytical methods, performs project audits, and ensures that data quality objectives are fulfilled.

**Field Team Leader and/or Field Hydrogeologist, Geologist or Engineer:** Implements the sampling program, supervises other sampling personnel, and ensures compliance with SOPs and QA/QC requirements. Prepares daily logs of field activities.

**Field Technician:** Responsible for sample collection, documentation, packaging, and shipping. Assists the FTL and/or geologist, hydrogeologist, or engineer in the implementation of tasks.

## 5.0 PROCEDURES

### 5.1 Applicability

The information in this SOP may be used by direct reference or incorporated into project-specific plans. Deviations or modifications to procedures addressed herein must be brought to the attention of, and approved by, applicable government agencies.

## 5.2 Sample Management

**Sample Containers:** The sample containers to be used will be dependent on the sample matrix and analyses desired, and are specified in the project FSP. Only certified pre-cleaned sample containers will be used. Sample containers will be filled with adequate headspace (approximately 10 percent) for safe handling upon opening, except containers for volatile organic compound (VOC) analyses, which will be filled completely with no headspace. This no-headspace requirement applies to both soil and groundwater samples.

Once opened, the containers will be used immediately. If the container is used for any reason in the field (e.g., screening) and not sent to the laboratory for analysis, it will be discarded. Prior to discarding the contents of the used container and the container, disposal requirements will be evaluated. When storing before and after sampling, the containers will remain separate from solvents and other volatile organic materials. Sample containers with preservatives added by the laboratory will not be used if held for an extended period on the job site or exposed to extreme heat conditions. Containers will be kept in a cool, dry place. For preserved samples (except VOCs), the pH of the sample will be checked following collection of the sample. If the pH is not at the required level, additional preservative (provided by the laboratory) will be added to the sample container.

**Numbering and Labeling:** Refer to OU3 SOP-9.

**Custody Seals.** Custody seals with the date and initials of the sampler will be used on each shipping container to ensure custody. The custody seal will be placed on opposite sides of the cooler across the seam of the lid and the cooler body. Alternatively, if the sample containers are all placed inside a liner bag within the cooler, the custody seal may be placed across the seal of the liner bag inside of the cooler.

**Chain-of-Custody:** COC procedures require a written record of the possession of individual samples from the time of collection through laboratory analyses. A sample is considered to be in custody if it is:

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- In a person's possession
- In view after being in physical possession
- In a secured condition after having been in physical custody
- In a designated secure area, restricted to authorized personnel

The COC record will be used to document the samples taken and the analyses requested. Refer to SOP-9 Attachment 2 for the OU3-specific COC form. Information recorded by field personnel on the COC record will include the following:

- Sample identifier (Index ID)
- Date and time of collection
- Sample matrix
- Preservation
- Type of analyses requested
- Unique COC number
- Lab being shipped to
- Signature of individuals involved in custody transfer (including date and time of transfer)
- Airbill number (if appropriate)
- Any comments regarding individual samples (e.g., organic vapor meter readings, special instructions).

COC records will be placed in a waterproof plastic bag (e.g., Ziploc®), taped to the inside lid of the cooler or placed at the top of the cooler, and transported with the samples. Signed airbills will serve as evidence of custody transfer between the field sampler and courier, as well as between the courier and laboratory. If a carrier service is used to ship the samples (e.g., Federal Express, etc.), custody will remain with the courier until it is relinquished to the laboratory. Upon receiving the sample cooler, a laboratory representative should sign in the receiving box of the COC, thus establishing custody. The sampler will retain copies of the COC record and airbill.

**Sample Preservation/Storage:** The requirements for sample preservation are dependent on the desired analyses and the sample matrix, and are specified in the FSP.

### 5.3 Sample Shipping

The methods and procedures described in this SOP were developed from these sources:

- 49 CFR 173. Shippers – Shippers – General Requirements for Shipping. United States Code of Federal Regulations available online at <http://www.gpoaccess.gov/cfr/index.html>
- 49 CFR 178. Specifications for Packaging. United States Code of Federal Regulations available online at <http://www.gpoaccess.gov/cfr/index.html>
- ASTM D 4220. Standard Practice for Preserving and Transporting Soil Samples. American Society for Testing and Materials available online at <http://www.astm.org/>
- ASTM D 4840. Standard Practice for Sampling Chain-of-Custody Procedures. American Society for Testing and Materials available online at <http://www.astm.org/>

Procedures for packaging and transporting samples to the laboratory are dependent on the chemical, physical, and hazard properties of the material. The procedures may also be based on an estimation of contaminant concentrations/properties in the samples to be shipped. Samples will be identified as environmental samples, excepted quantities samples, limited quantities samples, or standard hazardous materials. Environmental samples are defined as solid or liquid samples collected for chemical or geotechnical analysis. Excepted quantities involve the shipment of a few milliliters of either an acid or base preservative in an otherwise empty sample container. Limited quantities are restricted amounts of hazardous materials that may be shipped in generic, sturdy containers. Standard hazardous material shipments require the use of stamped/certified containers. All samples will be packaged and shipped or hand delivered to the laboratories the same day of sample collection, unless otherwise specified in the project-specific FSPs.

The following paragraphs describe standard shipping procedures for different types of samples. Any exceptions to these procedures will be defined in the FSP. It is the responsibility of the sampler to refer to the U.S. Department of Transportation (DOT) (<http://hazmat.dot.gov/regs/rules.htm>) regulations when dealing with a substance not addressed in this SOP for requirements and limitations associated with the shipment.

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**Sample Shipping via Commercial Carrier:**

**Aqueous or Solid Samples:** Samples will be packaged and shipped to the laboratories the same day of sample collection, unless otherwise specified in the FSP and depending on holding time requirements for individual samples. For aqueous or solid samples that are shipped to the laboratory via a commercial carrier the following procedures apply:

- Sample labels will be completed and attached to sample containers.
- The samples will be placed upright in a waterproof metal (or equivalent strength plastic) ice chest or cooler.
- For shipments containing samples for volatile organic analysis, include a trip blank.
- Ice in double Ziploc® bags (to prevent leakage) will be placed around, among, and on top of the sample bottles. Enough ice will be used so that the samples will be chilled and maintained at  $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$  during transport to the laboratory. Dry ice or blue ice will not be used.
- To prevent the sample containers from shifting inside the cooler, the remaining space in the cooler will be filled with inert cushioning material, such as shipping peanuts, additional bubble pack, or cardboard dividers, such that the sample containers remain upright and do not break.
- Tape shut the cooler's drain plug
- The original copy of the completed COC form will be placed in a waterproof plastic bag and taped to the inside of the cooler lid or placed at the top of the cooler.
- The lid will be secured by wrapping strapping tape completely around the cooler in two locations.

- Mark the cooler with arrow labels indicating the proper upright position of the cooler.
- Custody seals consisting of security tape with the date and initials of the sampler will be used on each shipping container to ensure custody. Two signed custody seals will be placed on the cooler, one on the front and one on the back.
- A copy of the COC record and the signed air bill will be retained for the project files.
- Affix a label containing the name and address of the shipper to the outside of the cooler

**Hand-Delivered Samples:** For aqueous or solid samples that will be hand carried to the laboratory, the same procedures apply.

**Excepted Quantities:** Usually, corrosive preservatives (e.g., hydrochloric acid, sulfuric acid, nitric acid, or sodium hydroxide) are added to otherwise empty sample bottles by the analytical laboratory prior to shipment to field sites. However, if there is an occasion whereby personnel are required to ship bottles with these undiluted acids or bases, the containers will be shipped in the following manner:

1. Each individual sample container will have not more than 30 milliliters of preservative.
2. Collectively, the preservative in these individual containers will not exceed a volume of 500 milliliters in the same outer box or package.
3. Despite the small quantities, only chemically compatible material may be placed in the same outer box, (e.g., sodium hydroxide, a base, must be packaged separately from the acids).
4. Federal Express will transport nitric acid only in concentrations of 40 percent or less.



5. A "Dangerous Goods in Excepted Quantities" label will be affixed to the outside of the outer box or container. Information required on the label includes:

- Signature of Shipper
- Title of Shipper
- Date
- Name and Address of Shipper
- Check of Applicable Hazard Class
- Listing of UN Numbers for Materials in Hazard Classes

**Limited Quantities:** Occasionally, it may become necessary to ship known hazardous materials, such as pure or floating product. DOT regulations permit the shipment of many hazardous materials in "sturdy" packages, such as an ice chest or cardboard box (not a specially constructed and certified container), provided the following conditions are met:

1. Each sample bottle is placed in a plastic bag, and the bag is sealed. Each VOC vial will be placed in a sealable bag. As much air as possible is squeezed from the bag before sealing. Bags may be sealed with evidence tape for additional security.
2. Or each bottle is placed in a separate paint can, the paint can is filled with vermiculite, and the lid is affixed to the can. The lid must be sealed with metal clips, filament, or evidence tape. If clips are used, the manufacturer typically recommends six clips.
3. The cans are placed upright in a cooler that has had the drain plug taped shut inside and outside, and the cooler is lined with a large plastic bag. Approximately 1 inch of adsorbent material sufficient to retain any liquid that may be spilled, is placed in the bottom of the liner. Only containers having chemically compatible material may be packaged in each cooler or other outer container.
4. The COC record is sealed inside a plastic bag and placed inside the cooler. The sampler retains one copy of the COC record. The laboratory will be notified if the

sample is suspected of containing any substance for which the laboratory personnel should take safety precautions.

5. The cooler is shut and sealed with strapping tape (filament type) around both ends. Two signed custody seals will be placed on the cooler, one on the front and one on the back. Additional seals may be used if the sampler and/or shipper consider more seals to be necessary. Wide, clear tape will be placed over the seals to ensure against accidental breakage.
6. The following markings are placed on the side of the cooler:
  - Proper Shipping Name (Column B, List of Dangerous Goods, Section 4, IATA Dangerous Goods Regulations [DGR])
  - UN Number (Column A, List of Dangerous Goods, Section 4, IATA DGR)
  - Shipper's name and address
  - Consignee's name and address
  - The words "LIMITED QUANTITY"
  - Hazard Labels (Column E, List of Dangerous Goods, Section 4, IATA DGR)
  - Two Orientation (Arrow) labels placed on opposite sides.
7. The Airbill/Declaration of Dangerous Goods form is completed as follows:
  - Shipper's name and address
  - Consignee's name and address
  - Services, Delivery & Special Handling Instructions
  - Cross out "Cargo Aircraft Only" in the Transport Details Box
  - Cross out "Radioactive" under Shipment Type
  - Nature and Quantity of Dangerous Goods

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- Proper Shipping Name (Column B, List of Dangerous Goods, Section 4, IATA DGR)
- Class or Division (Column C, List of Dangerous Goods, Section 4, IATA DGR)
- UN Number (Column A, List of Dangerous Goods, Section 4, IATA DGR)
- Packing Group (Column F, List of Dangerous Goods, Section 4, IATA DGR)
- Subsidiary Risk, if any (Column D, List of Dangerous Goods, Section 4, IATA DGR)
- Quantity and type of packing (number and type of containers: for example, "3 plastic boxes", and the quantity per container, "2 L", is noted as "3 Plastic boxes X 2 L" This refers to 3 plastic boxes (coolers are referred to as plastic boxes) with 2 liters in each box.
- Packing Instructions (Column G, List of Dangerous Goods, Section 4, IATA DGR).
- Note: Only those Packing Instructions in Column G that begin with the letter "Y" may be used. These refer specifically to the Limited Quantity provisions.
- Authorization (Write in the words Limited Quantity)
- Emergency Telephone Number (List 800-535-5053. This is the number for INFOTRAC.)
- Printed Name and Title, Place and Date, Signature.

**Standard Hazardous Materials:** Shipment of standard hazardous materials presents the most difficulty and expense. However, there may be occasion whereby a hazardous material cannot be shipped under the Limited Quantity provisions, (e.g., where there is no Packing Instruction in Column G, List of Dangerous Goods, IATA Dangerous Goods Regulations, that is preceded by the letter "Y").

In such cases, the general instructions noted above but for non-Limited Quantity materials will apply, with one important difference: standard hazardous materials shipment requires the use of certified outer shipping containers. These containers have undergone rigid testing and are, therefore, designated by a "UN" stamp on the outside, usually along the bottom of a container's side. The UN stamp is also accompanied by codes specifying container type, packing group rating, gross mass, density, test pressure, year of manufacturer, state of manufacturer, and manufacturer code name. The transport of lithium batteries in Hermit Data Loggers is an example of a standard hazardous material where only a designated outer shipping container may be used.

#### **5.4 Holding Times**

The holding times for samples will depend on the analysis and the sample matrix. Refer to the FSP for holding times requirements.

### **6.0 QUALITY ASSURANCE AND QUALITY CONTROL**

All sample shipments must be documented in the field logbooks and/or field forms, including rationales deviations from this SOP. The Field Team Leader or designated QA reviewer will check and verify that handling and shipment documentation has been completed per this procedure and other procedures referenced herein.

### **7.0 DECONTAMINATION**

All shipment coolers shall be maintained clean of sampled material to avoid exposure during shipment. Any investigation-derived waste generated in the sampling process shall be managed in accordance with the procedures outlined in SOP-12.

### **8.0 REFERENCES**

Agency for Toxic Substances and Disease Registry. 2006. Asbestos Exposure and Your Health.

Libby Superfund Site Operable Unit 3 Standard Operating Procedure

Enforcement Considerations for Evaluations of Uncontrolled Hazardous Waste Disposal Sites by Contractors, Draft, Appendix D, April 1980.

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Libby Superfund Site Operable Unit 3 Standard Operating Procedure

Date: May 20, 2009

OU3 SOP 9 (Rev. 5)

Title: FIELD DOCUMENTATION

**APPROVALS:**

TEAM MEMBER

SIGNATURE/TITLE

DATE

EPA Remedial Project Manager

*[Signature]* / RPM

5/21/09

SOP Author

*[Signature]*

5/22/09

Revision Number	Date	Reason for Revision
0	09/26/2007	--
1	10/5/2007	<ul style="list-style-type: none"> <li>Add section for "Corrections and Modifications" and Field Modification Approval form (Attachment 3)</li> <li>Update Labeling section and COC (Attachment 2) to reflect non-asbestos analysis and container details</li> <li>Update FSDS forms (Attachment 1) based on field team input</li> </ul>
2	02/22/2008	<ul style="list-style-type: none"> <li>Incorporate changes to FSDS forms (Attachment 1) based on field input</li> <li>Remove OU3 phase specificity in SOP text</li> </ul>
3	05/29/2008	<ul style="list-style-type: none"> <li>Incorporate changes to FSDS forms (Attachment 1) based on field input</li> </ul>
4	06/30/2008	<ul style="list-style-type: none"> <li>Update Attachment 1 with all OU3 FSDS forms (including those used in Phase I and Phase II)</li> <li>Remove OU3 phase specificity in Attachments</li> </ul>
5	05/20/2009	<ul style="list-style-type: none"> <li>Add FSDS form for ABS Personal Air</li> <li>Add FSDS form for Small Mammal Tissue</li> <li>Modified COC to change medium code to "A-Air" to accommodate both ambient and activity-based sampling (ABS) air samples</li> <li>Added new media code for small mammal tissue "MT"</li> </ul>

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Rev. 5

Date: May 20, 2009

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## 1.0 INTRODUCTION

This Standard Operating Procedure (SOP) is a general guidance document for the required documentation to be completed by field personnel during field investigations. This SOP is based on MWH SOP-04, Field Documentation, Revision 1.0, March 2006, modified for use at the Libby Mine Site. Documentation in the form of field logbooks, reports, and forms shall be completed for every activity in the field. Records shall be maintained on a daily basis as the work progresses. All field documentation shall be accurate and legible because it is deliverable to the client as potentially a legal document.

## 2.0 HEALTH AND SAFETY WARNING

All personnel engaged in soil sampling must follow health and safety protocols described in the site health and safety plan. Asbestos fibers are thin and long fibers so small that they cannot be seen by the naked eye. Asbestos fibers are easily inhaled when disturbed and when embedded in the lung tissue can cause health problems. Significant exposure to asbestos increases the risk of lung cancer, mesothelioma, asbestosis (non-cancerous lung disease), and other respiratory diseases (ATSDR 2006). All personnel engaged in soil sampling must follow health and safety protocols described in the health and safety plan.

## 3.0 RESPONSIBILITIES

This section presents a brief definition of field roles, and the responsibilities generally associated with them. This list is not intended to be comprehensive and often, additional personnel may be involved. Project team member information shall be included in project-specific plans (e.g., work plan, field sampling plan, quality assurance plan, etc.), and field personnel shall always consult the appropriate documents to determine project-specific roles and responsibilities. In addition, one person may serve in more than one role on any given project.

**Project Manager:** Selects project-specific field documentation with input from other key project staff.



**Quality Control Manager:** Overall management and responsibility for quality assurance and quality control (QA/QC). Selects QA/QC procedures for the sampling and analytical methods, performs project audits, and ensures that data quality objectives are fulfilled.

**Field Team Leader (FTL) and/or Field Geologist, Hydrogeologist, or Engineer:** Implements the sampling program, supervises other sampling personnel, and ensures compliance with SOPs and QA/QC requirements. Prepares daily logs of field activities.

**Field Technician (or other designated personnel):** Assists the FTL and/or field geologist, hydrogeologist, or engineer in the implementation of field tasks and field documentation.

**Field Sample/Data Manager:** Responsible for proper handling and shipping of all samples collected by the field crew, electronic data entry of field sample data sheet (FSDS) and chain-of-custody (COC) forms, and scanning/posting of field documentation PDFs (FSDS, COC, field logbooks, digital photographs) to a dedicated FTP site.

#### **4.0 FIELD DOCUMENTATION PROCEDURES**

Field documentation serves as the primary foundation for all field data collected that will be used to evaluate the project site. There are two main forms of field documentation – field logbooks and FSDS forms. All field documentation shall be accurate, legible and written in indelible black or blue ink. Absolutely no pencils or erasures shall be used. Incorrect entries in the FSDS forms or field logbooks will be corrected by crossing out the incorrect entry with one line, the individual making the correction will initial and date next to the correction.

##### **4.1 Field Logbooks**

The field logbook shall be a bound, weatherproof book with numbered pages, and shall serve primarily as a daily log of the activities carried out during the fieldwork. All entries shall be made in indelible black or blue ink. A field logbook shall be completed for each operation undertaken during the field tasks. To further assist in the organization of the field log books, the project name and the date shall be recorded on top of each page along with the significant

activity description (e.g., surface sample or soil boring number). All original field documentation shall be retained in the project files.

Skipped pages or blank sections at the end of a field log book page shall be crossed out with an "X" covering the entire page or blank section; "No Further Entries," initials, and date shall be written by the person crossing out the blank section or page. The responsible field team member shall write his/her signature, date, and time after the day's last entry.

Field activities vary from project to project; however, the concept and general information that shall be recorded are similar. The descriptions of field data documentation given below serve as an outline; individual activities may vary in documentation requirements. A detailed description of two basic example logbooks, suitable for documentation of field activities, is given below. These field logbooks include the FTL logbook and the field geologist/sampling team logbook.

**FTL Logbook:** The FTL's responsibilities include the general supervision, support, assistance, and coordination of the various field activities. As a result, a large portion of the FTL's day is spent rotating between operations in a supervisory mode. Records of the FTL's activities, as well as a summary of the field team(s) activities, shall be maintained in a logbook. The FTL's logbook shall be used to fill out daily/weekly reports and daily quality control reports (DQCRs), and therefore, shall contain all required information. Entries shall be preceded with time in military units for each observation. Items to be documented include:

- Record of tailgate meetings
- Personnel and subcontractors on job site and time spent on the site
- Field operations and personnel assigned to these activities
- Site visitors
- Log of FTL's activities: time spent supervising each operation and summary of daily operations as provided by field team members
- Problems encountered and related corrective actions
- Deviations from the sampling plan and reasons for the deviations
- Records of communications; discussions of job-related activities with the client, subcontractor, field team members, and project manager

- Information on addresses and contacts
- Record of invoices signed and other billing information
- Field observations

**Field Geologist/Sampling Team Logbook:** The field geologist or sampling team leader shall be responsible for recording the following information in a logbook:

- Health and Safety Activities
  - Calibration records for health and safety equipment (e.g., type of PID, calibration gas used, associated readings, noise dosimeters, etc.)
  - Personnel contamination prevention and decontamination procedures
  - Record of daily tailgate safety meetings
- Weather
- Calibration of field equipment
- Equipment decontamination procedures
- Personnel and subcontractors on job site and time spent on the site
- Station identifier
- Sampling activities
  - Sample location (sketch)
  - Equipment used
  - Names of samplers
  - Date and time of sample collection
  - Sample interval
  - Number of samples collected
  - Analyses to be performed on collected samples
- Disposal of contaminated wastes (e.g., PPE, paper towels, Visqueen, etc.)
- Field observations
- Problems encountered and corrective action taken
- Deviations from the sampling plan and reason for the deviations
- Site visitors

## 4.2 Field Sample Documentation

**Sample Labels:** A unique sample identification label shall be affixed to all sample containers. All samples will be labeled in a clear, precise way for proper identification in the field and for tracking in the laboratory. At the time of collection, each sample will be labeled with a unique 5-digit sequential identification (ID) number, referred to as the Index ID. The Index ID for all samples collected as part of OU3 sampling activities will have a two-character prefix specific to the sampling Phase (e.g., Phase 1 samples will have a "P1" prefix, P1-12345) as specified in the applicable SAP. Index ID labels will be ½ inch x 1 ¾ inch in size and pre-printed for use in the field. For each Index ID, multiple labels will be printed to allow for multiple containers of the same sample (i.e., for different analyses).

Index ID Label Example:

**P1-12345**

Each collection container will be labeled with a container label that enables the field team member to record the container-specific details, such as the method of sample preparation (e.g., filtered/unfiltered), method of preservation, and the analytical methods that will be requested. Container labels will be 2 inch x 4 inch in size and pre-printed for use in the field. Any container-specific information shall be written in indelible ink.

Container Label Example:

Index ID:		Date/Time: _____	
Media (circle one):    AQ    SO    A    BK    DB    TC    MT			
For AQ, Filtered? (circle one):    Yes    No			
Container: _____			
Preservation: _____			
Analyses: _____			
_____			

Media acronyms: AQ – aqueous media, SO – solid media, A – air,  
BK – tree bark, DB – organic debris, TC – tree age core, MT – mammal tissue

After labels have been affixed to the sample container, the labels will be covered with clear packaging tape to ensure permanence during shipping.

Any unused Index ID labels should be crossed out to avoid the possibility of using unused labels for a different sample.

**Field Sample Data Sheet (FSDS) Forms:** Data regarding each sample collected as part of the OU3 sampling will be documented using Libby-specific FSDS forms (provided as Attachment 1). These FSDS forms are medium-specific and designed to facilitate data entry of station location, sample details, and field measurements needed for the OU3 investigation.

In the field, one field team member will be responsible for recording all sample details onto the appropriate FSDS form. At the time of sample labeling, one Index ID label will be affixed to the FSDS form in the appropriate field. All written entries on the FSDS form shall be accurate, legible and written in indelible black or blue ink.

Once the FSDS form is complete, written entries will be checked by a second field team member. These two field team members will initial the bottom of the FSDS form in the appropriate field to document who performed the written data entry and who performed the QC check of the FSDS form.

On a weekly basis (or more frequently as conditions permit), information from the hard copy FSDS form will be manually entered into a field-specific OU3 database using electronic data entry screens by the Field Sample/Data Manager. Once electronic data entry is complete, QC of all data entry will be completed by the FTL or their designate. The Field Sample/Data Manager and the FTL will initial in the appropriate field on the paper FSDS form to document who performed the data entry into the database and who performed the QC check.

#### **4.3 Photologs**

Photologs are often used in the field to document site conditions and sample location characteristics. While photographs may not always be required, they shall be used wherever

applicable to show existing site conditions at a particular time and stage of the investigation or related site activity. Photolog information shall include:

- station location identifier
- Index ID (if applicable)
- date and time of photo
- direction/orientation of the photo
- description of what the photo is intended to show

An engineer's scale or tape shall be included in any photographs where scale is necessary. Any wasted frames or images in a roll of film or sequence of digital images shall be so noted in the field logbook.

#### **4.4 Chain-of-Custody Records**

**Custody Seals:** Custody seals with the date and initials of the sampler will be used on each shipping container to ensure custody. The custody seal will be placed on opposite sides of the cooler across the seam of the lid and the cooler body. Alternatively, if the sample containers are all placed inside a liner bag within the cooler, the custody seal may be placed across the seal of the liner bag inside of the cooler.

**Chain-of-Custody Forms:** COC procedures allow for the tracking of possession and handling of individual samples from the time of field collection through to laboratory analysis. Documentation of custody is accomplished through a COC form that lists each sample and the individuals responsible for sample collection and shipment, sample preparation, and receipt by the analytical laboratory. The COC form also documents the analyses requested for each sample. Whenever a change of custody takes place, both parties will sign and date the COC form, with the relinquishing party retaining a copy of the form. The party that accepts custody will inspect the COC form and all accompanying documentation to ensure that the information is complete and accurate. Any discrepancies will be noted on the COC form. Shipping receipts shall be signed and filed as evidence of custody transfer between field sampler(s), courier, and laboratory.

Attachment 2 provides an example of the COC form that will be used for all samples collected as part of OU3 sampling. This form will be printed as a carbonless triplicate form to facilitate retention of COC copies by relinquishing parties. As seen, the COC form includes the following information:

- sample identifier (Index ID)
- date and time of collection
- method of sample preparation and preservation
- number of sample containers
- analyses requested
- shipping arrangements and airbill number, as applicable
- recipient laboratories
- signatures of parties relinquishing and receiving the sample

On a daily basis, the Field Sample/Data Manager will package samples for shipping, complete hard copy COC forms, and ship all samples as outlined in SOP No. 8. On a daily basis, information from the hard copy COC form necessary for sample tracking will be manually entered into a field-specific OU3 database using electronic data entry screens by the Field Sample/Data Manager. Once electronic data entry is complete, QC of all data entry will be completed by the FTL or their designate.

## 5.0 FIELD DATA TRANSMITTAL

Copies of all FSDS forms, COC forms, and field log books will be scanned and posted in portable document format (PDF) to a project-specific file transfer protocol (FTP) site daily. This FTP site will have controlled access (i.e., user name and password are required) to ensure data access is limited to appropriate project-related personnel. File names for scanned FSDS forms, COC forms, and field log books will include the sample date in the format YYYYMMDD to facilitate document organization (e.g., FSDS\_20090831.pdf).

Electronic copies of all digital photographs will also be posted weekly (or more frequently as conditions permit) to the project-specific FTP site. File names for digital photographs will include the station identifier, the sample date, and photograph identifier (e.g., ST-1\_20090831\_12459.tif).

A copy of the field-specific OU3 database will be posted to the project-specific FTP site on a weekly basis (or more frequently as conditions permit). The field-specific OU3 database posted to the FTP site will include the post date in the file name (e.g., FieldOU3DB\_20090831.mdb).

## **6.0 CORRECTIONS AND MODIFICATIONS**

### **6.1 Field Deviations and Modifications**

It is recognized that deviations and modifications from the standard operating procedures may be necessary based on site conditions. Any requested field modifications will be submitted by Robert Marriam (Remedium Group, Inc. - W.R. Grace contractor) to Bonita Lavelle (EPA Region 8 - Remedial Project Manager) for review and approval. All modification requests will be recorded in a Field Modification Approval Form (see Attachment 3).

### **6.2 Corrections to Hard Copy Forms**

If an error is identified on an FSDS or COC form prior to entry into the field-specific OU3 database, the information should be corrected on the hard copy form by crossing out the incorrect entry with one line, the individual making the correction will initial and date next to the correction. Data entry into the field-specific OU3 database and scanning/posting of the hard copy forms should proceed following the data entry procedures described above.

If an error is identified on an FSDS or COC form after entry into the field-specific OU3 database, the information should be corrected on the hard copy form by crossing out the incorrect entry with one line, the individual making the correction will initial and date next to the correction. The corrected form should be scanned and posted to the project-specific FTP site. File names for corrected FSDS forms will include the Index ID of the corrected sample to facilitate document organization (e.g., FSDS\_C\_P1-12345.pdf). File names for corrected COC



forms will include the COC ID of the corrected COC form to facilitate document organization (e.g., COC\_C\_OU3-36512.pdf). Necessary data corrections will be made to the master OU3 database by the database manager.

If changes are made to a COC form, the analytical laboratory should be provided with a corrected COC form.

## 7.0 REFERENCES

Agency for Toxic Substances and Disease Registry. 2006. Asbestos Exposure and Your Health.

RCRA Ground-Water Monitoring: Draft Technical Guidance, November 1992.

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**ATTACHMENT 1**

**OU3 FIELD SAMPLE DATA SHEET (FSDS) FORMS**

*This page intentionally left blank to facilitate double-sided printing.*

# LIBBY OU3 FIELD SAMPLE DATA SHEET (FSDS) rev2

## STATIONARY AMBIENT AIR MONITOR

Field Logbook No: \_\_\_\_\_ Page No: \_\_\_\_\_

☐ Check box if GPS information  
has been recorded previously

Station ID: \_\_\_\_\_ Station Comments: \_\_\_\_\_

GPS Coordinate System: UTM Zone 11 North, NAD83 datum, meters

X coord: \_\_\_\_\_ Y coord: \_\_\_\_\_ Elevation: \_\_\_\_\_ m

Sampling Team: \_\_\_\_\_ Sampler Initials: \_\_\_\_\_

Data Item	Cassette 1	Cassette 2	Cassette 3
Index ID	AFFIX LABEL HERE	AFFIX LABEL HERE	AFFIX LABEL HERE
Sample Height (ft)			
Location Description			
Field QC Type (circle)	FS-(field sample) FB-(field blank) FD-(field dup) For FD, Parent ID:	FS-(field sample) FB-(field blank) FD-(field dup) For FD, Parent ID:	FS-(field sample) FB-(field blank) FD-(field dup) For FD, Parent ID:
Matrix Type	Outdoor	Outdoor	Outdoor
Flow Meter Type	Rotameter	Rotameter	Rotameter
Archive blank (circle)	Yes No	Yes No	Yes No
Pump ID Number			
Flow Meter ID Number			
Start Date (mm/dd/yy)			
Start Time (hh:mm)			
Start Counter			
Daily Flow Check:	Check1 Time Flow	Check1 Time Flow	Check1 Time Flow
Record time (hh:mm) and flow rate (L/min) in fields provided	Check2	Check2	Check2
	Check3	Check3	Check3
	Check4	Check4	Check4
Stop Date (mm/dd/yy)			
Stop Time (hh:mm)			
Stop Counter			
Pump fault? (circle)	Yes No	Yes No	Yes No
Stop Flow (L/min)			
Field Comments			
Cassette Lot Number:			
Entered By (Provide initials):	Validated By (Provide initials):		

For Data Entry Completion (Provide Initials)

Completed by:

QC by:

# LIBBY OU3 FIELD SAMPLE DATA SHEET (FSDS) rev2

## FOREST SOIL AND TREE BARK

Field Logbook No: \_\_\_\_\_

Page No: \_\_\_\_\_

Station ID: \_\_\_\_\_

Sampling Date: \_\_\_\_\_

GPS Coordinate System: UTM Zone 11 North, NAD83 datum, meters

X coord: \_\_\_\_\_ Y coord: \_\_\_\_\_ Elevation: \_\_\_\_\_ m

Sampling Team: \_\_\_\_\_ Sampler Initials: \_\_\_\_\_

Station Comments: \_\_\_\_\_

### TREE BARK SAMPLES

Index ID: _____	Field QC Type (circle one): FS (field sample) FD (field duplicate) For FD, Parent ID: _____	Sample Area (cm <sup>2</sup> ): _____	Tree Species: _____  Collection Height (ft): _____ Diameter* (in): _____	Age Core Collected? (circle one): Y N
Index ID: _____	Field QC Type (circle one): FS (field sample) FD (field duplicate) For FD, Parent ID: _____	Sample Area (cm <sup>2</sup> ): _____		
Field Comments: _____				
Entered by (Provide initials): _____			Validated by (Provide initials): _____	

\*Measured with "D-tape"

### FOREST SOIL SAMPLES

Index ID: _____	Field QC Type (circle one): FS (field sample) FD (field duplicate) For FD, Parent ID: _____	Bulk Soil Description		Organic Debris Collected? (circle one): Y N
		Depth (in) Start: _____ End: _____	Sample Type: Grab Composite # of Comp.: _____	
Index ID: _____	Field QC Type (circle one): FS (field sample) FD (field duplicate) For FD, Parent ID: _____	Bulk Soil Description		Organic Debris Collected? (circle one): Y N
		Depth (in) Start: _____ End: _____	Sample Type: Grab Composite # of Comp.: _____	
Index ID: _____	Field QC Type (circle one): FS (field sample) FD (field duplicate) For FD, Parent ID: _____	Bulk Soil Description		Organic Debris Collected? (circle one): Y N
		Depth (in) Start: _____ End: _____	Sample Type: Grab Composite # of Comp.: _____	
Field Comments: _____				
Entered by (Provide initials): _____			Validated by (Provide initials): _____	

For Data Entry Completion (Provide Initials)

Completed by

QC by

For Data Entry Completion (Provide Initials)	Completed by	QC by
----------------------------------------------	--------------	-------

# LIBBY OU3 FIELD SAMPLE DATA SHEET (FSDS) rev2

## SOIL-LIKE MATERIALS

Field Logbook No: \_\_\_\_\_ Page No: \_\_\_\_\_

Station ID: \_\_\_\_\_ Sampling Date: \_\_\_\_\_

GPS Coordinate System: UTM Zone 11 North, NAD83 datum, meters

Sampling Team: \_\_\_\_\_ Sampler Initials: \_\_\_\_\_

Station Comments: \_\_\_\_\_

Data Item	Sample 1	Sample 2	Sample 3
Index ID	AFFIX LABEL HERE	AFFIX LABEL HERE	AFFIX LABEL HERE
Matrix (circle one):	Surface Soil    Tailings Waste Rock    Roadway Other _____	Surface Soil    Tailings Waste Rock    Roadway Other _____	Surface Soil    Tailings Waste Rock    Roadway Other _____
Sample Time (hh:mm)			
Sample Type (circle one):	Grab    Composite # of Comp: _____	Grab    Composite # of Comp: _____	Grab    Composite # of Comp: _____
Sample Depth	Start Depth (in): _____ End Depth (in): _____	Start Depth (in): _____ End Depth (in): _____	Start Depth (in): _____ End Depth (in): _____
Field QC Type (circle one):	FS (field sample) FD (field duplicate) For FD, Parent ID: _____ TB (trip blank) Cooler: _____ PE (perf. eval.) ID: _____	FS (field sample) FD (field duplicate) For FD, Parent ID: _____ TB (trip blank) Cooler: _____ PE (perf. eval.) ID: _____	FS (field sample) FD (field duplicate) For FD, Parent ID: _____ TB (trip blank) Cooler: _____ PE (perf. eval.) ID: _____
Transect Start Location or Grab Sample Location	X coord: _____ m Y coord: _____ m Elevation: _____ m	X coord: _____ m Y coord: _____ m Elevation: _____ m	X coord: _____ m Y coord: _____ m Elevation: _____ m
Transect End Location	X coord: _____ m Y coord: _____ m Elevation: _____ m	X coord: _____ m Y coord: _____ m Elevation: _____ m	X coord: _____ m Y coord: _____ m Elevation: _____ m
Field Comments:			
Cooler:			
Entered by (Provide initials):	Validated by (Provide initials):		

For Data Entry Completion (Provide Initials)

Completed by

QC by



# LIBBY OU3 FIELD SAMPLE DATA SHEET

## SURFACE WATER AND SEDIMENT

Station ID: \_\_\_\_\_ Sampling Date: \_\_\_\_\_  
 Field Logbook ID: \_\_\_\_\_ Logbook Page No: \_\_\_\_\_  
 GPS Coordinate System: UTM Zone 11 North, NAD83 datum, meters  
**For New Stations Only:** X coord: \_\_\_\_\_ Y coord: \_\_\_\_\_ Elev: \_\_\_\_\_  
 Sampling Team: MWH Samplers Initials: \_\_\_\_\_

### WATER QUALITY PARAMETERS (if applicable)

Time Measured (hh:mm)	Temp. (°C)	pH	Specific Conductance (mS/cm Auto-comp @ 25°C)	Diss. O <sub>2</sub> (mg/L)	ORP (mV)	Turbidity (NTU)

### SAMPLE COLLECTION

Index ID	AFFIX LABEL HERE	Sampling Time: _____ Sample Type: Field Sample  Media : Surface Water Sediment	Sampling Method (if applicable): Grab or Composite # of Composites: _____ Sampling Depth: Top (in) _____ Bot (in) _____
Index ID	AFFIX LABEL HERE	Sampling Time: _____ Sample Type: SP FD MS MSD PE FB TB EB Media : Surface Water Sediment	Sampling Method (if applicable): Grab or Composite # of Composites: _____ Sampling Depth: Top (in) _____ Bot (in) _____
Index ID	AFFIX LABEL HERE	Sampling Time: _____ Sample Type: SP FD MS MSD PE FB TB EB Media : Surface Water Sediment	Sampling Method (if applicable): Grab or Composite # of Composites: _____ Sampling Depth: Top (in) _____ Bot (in) _____
Index ID	AFFIX LABEL HERE	Sampling Time: _____ Sample Type: SP FD MS MSD PE FB TB EB Media : Surface Water Sediment	Sampling Method (if applicable): Grab or Composite # of Composites: _____ Sampling Depth: Top (in) _____ Bot (in) _____

### COMMENTS

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Note: FS Field Sample SP Field Split Sample FD Field Duplicate Sample  
 TB Trip Blank Sample MS Matrix Spike Sample MSD Matrix Spike Duplicate Sample  
 FB Field Blank Sample EB Equipment Decon Blank Sample PE Performance Evaluation Sample

Field Data Entered by: \_\_\_\_\_ Field Entries Checked by: \_\_\_\_\_

Database Entry: \_\_\_\_\_

Database QC: \_\_\_\_\_

# LIBBY OU3 FIELD SAMPLE DATA SHEET (FSDS) SMALL MAMMAL TISSUE COLLECTION

Field Logbook ID: \_\_\_\_\_ Logbook Page No: \_\_\_\_\_

Necropsy Date: \_\_\_\_\_ Personnel Initials: \_\_\_\_\_

Small Mammal Field ID: SM- \_\_\_\_\_ - \_\_\_\_\_ - \_\_\_\_\_ Animal Weight (grams): \_\_\_\_\_ (w/o fetuses if pregnant)  
[SM - station ID - transect ID - trap# - animal#]

General Necropsy Comments: \_\_\_\_\_

	TISSUE #1				TISSUE #2				TISSUE #3				TISSUE #4			
<b>Tissue Type (circle one):</b>	TY	AR	ES		TY	AR	ES		TY	AR	ES		TY	AR	ES	
	ST	SIN	LIN		ST	SIN	LIN		ST	SIN	LIN		ST	SIN	LIN	
	LU	EY	CAR		LU	EY	CAR		LU	EY	CAR		LU	EY	CAR	
	Other: _____				Other: _____				Other: _____				Other: _____			
<b>Weight (mg):</b>																
<b>Index ID:</b>	Affix Label Here				Affix Label Here				Affix Label Here				Affix Label Here			
<b>Field QC Type (circle one):</b>	FS	FD	TB		FS	FD	TB		FS	FD	TB		FS	FD	TB	
<b>Tissue Comments:</b>																

	TISSUE #5				TISSUE #6				TISSUE #7				TISSUE #8			
<b>Tissue Type (circle one):</b>	TY	AR	ES		TY	AR	ES		TY	AR	ES		TY	AR	ES	
	ST	SIN	LIN		ST	SIN	LIN		ST	SIN	LIN		ST	SIN	LIN	
	LU	EY	CAR		LU	EY	CAR		LU	EY	CAR		LU	EY	CAR	
	Other: _____				Other: _____				Other: _____				Other: _____			
<b>Weight (mg):</b>																
<b>Index ID:</b>	Affix Label Here				Affix Label Here				Affix Label Here				Affix Label Here			
<b>Field QC Type (circle one):</b>	FS	FD	TB		FS	FD	TB		FS	FD	TB		FS	FD	TB	
<b>Tissue Comments:</b>																

	TISSUE #9				TISSUE #10				TISSUE #11				TISSUE #12			
<b>Tissue Type (circle one):</b>	TY	AR	ES		TY	AR	ES		TY	AR	ES		TY	AR	ES	
	ST	SIN	LIN		ST	SIN	LIN		ST	SIN	LIN		ST	SIN	LIN	
	LU	EY	CAR		LU	EY	CAR		LU	EY	CAR		LU	EY	CAR	
	Other: _____				Other: _____				Other: _____				Other: _____			
<b>Weight (mg):</b>																
<b>Index ID:</b>	Affix Label Here				Affix Label Here				Affix Label Here				Affix Label Here			
<b>Field QC Type (circle one):</b>	FS	FD	TB		FS	FD	TB		FS	FD	TB		FS	FD	TB	
<b>Tissue Comments:</b>																

Tissue Type Descriptors: TY = thyroid; AR = adrenal gland; ES = esophagus; SIN = small intestine; LIN = large intestine; LU = lung; EY = eyeball; CAR = carcass  
Field QC Type Descriptors: FS = Field Sample; FD = Field Duplicate; TB = Tissue Blank

For Data Entry Completion (Provide Initials)

Completed by

QC by

**ATTACHMENT 2**

**OU3 CHAIN OF CUSTODY FORM**

## COC No. \_\_\_\_\_

PAGE: OF:

ENTERED BY (Signature): \_\_\_\_\_ PROJECT MANAGER: \_\_\_\_\_ DATE: \_\_\_\_\_

METHOD OF SHIPMENT:		CARRIER/WAYBILL NO.:	DESTINATION:
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[illegible]

			TOTAL NUMBER OF CONTAINERS	LABORATORY COMMENTS/CONDITION OF SAMPLES			Cooler Temp:
RELINQUISHED BY:			DATE	TIME	RECEIVED BY:		
SIGNATURE	PRINTED NAME	COMPANY			SIGNATURE	PRINTED NAME	COMPANY

(a) Method, container, and preservation details are provided in the attached tables  
(b) With Libby-specific modifications. See applicable O3 SAP for counting and stopping rules  
(c) See applicable SAP for details on preparation methods.

(d) Preparation by ISSI-LIBBY-01 and analysis by SRC-LIBBY-01 (PLM-Grav) and SRC-LIBBY-03 (PLM-VE)  
(e) In accordance with procedures in Phipps (1985).

**DISTRIBUTION:** PINK: Field Copy YELLOW: Laboratory Copy WHITE: Return to Originator

**ATTACHMENT 3**

**OU3 FIELD MODIFICATION APPROVAL FORM**

# FIELD MODIFICATION APPROVAL FORM

LFM-OU3-\_\_\_\_\_

*Libby OU3 Phase \_\_\_\_ Sampling & Analysis Plan*

Requested by: \_\_\_\_\_ Date: \_\_\_\_\_

Description of Deviation:

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☐ EPA Region 8 has reviewed this field modification approves as proposed.

☐ EPA Region 8 has reviewed this field modification and approves with the following exceptions:

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☐ EPA Region 8 has reviewed this field modification and does not agree with the proposed approach for the following reasons:

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\_\_\_\_\_  
Bonita Lavelle, EPA RPM

\_\_\_\_\_  
Date

**Libby Superfund Site Operable Unit 3 Standard Operating Procedure**

Date: September 26, 2007

OU3 SOP 12 (Rev. 0)

Title: INVESTIGATION DERIVED WASTE (IDW) MANAGEMENT

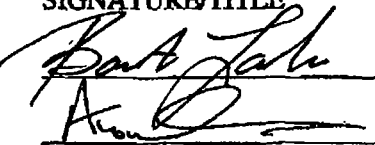
**APPROVALS:**

TEAM MEMBER

SIGNATURE/TITLE

DATE

EPA Remedial Project Manager



9/26/07

SOP Author



9/26/07

Revision Number	Date	Reason for Revision
0	09/26/2007	--

## 1.0 INTRODUCTION

This SOP is based on MWH SOP-07, Investigation-Derived Waste (IDW) Management, Revision 1.0, April 2007, modified for use at the Libby Asbestos Superfund Site OU3. IDW may be generated during field investigations at the Libby Asbestos Superfund Site OU3. The National Contingency Plan (NCP), codified in 40 Code of Federal Regulations (CFR) 300, requires that IDW be handled to attain all the applicable or relevant and appropriate requirements (ARARs) to the extent practicable, considering the urgency of the situation. The purpose of this SOP is to present procedures to be followed in the management of IDW generated during the field investigations.

Typical IDW generated during field activities are solid wastes and may include (but are not limited to) the following media and waste types:

Fluids	Solids
Purge water and groundwater	Soils and soil cuttings
Drilling mud	Plastic tarps or sheeting
Grout	Drill pipe and well casing/screen
Decontamination fluids and wastewater	Decontamination solids
	Disposable equipment (i.e., rope, bailers, sampling equipment, & other consumables)
	Spent personal protective equipment (PPE)
	Used containers, sample bottles
	Packaging materials

The above wastes may or may not be encountered, generated or managed while performing field investigations. However, all solid waste streams will be characterized to determine if they are hazardous wastes per 40 CFR § 262.11 for the purposes of handling and disposal. Guidance from this document shall be used as part of project planning to estimate total volumes of IDW likely to be generated as well as how the IDW will be managed and disposed.



## 2.0 HEALTH AND SAFETY WARNING

All personnel engaged in IDW handling must follow health and safety protocols described in the health and safety plan. Asbestos fibers are thin and long fibers so small that they cannot be seen by the naked eye. Asbestos fibers are easily inhaled when disturbed and when embedded in the lung tissue can cause health problems. Significant exposure to asbestos increases the risk of lung cancer, mesothelioma, asbestosis (non-cancerous lung disease), and other respiratory diseases (ATSDR 2006).

## 3.0 DEFINITIONS

**Area of Contamination (AOC) unit:** The AOC unit concept is critical to the IDW management at a CERCLA investigation site. Although EPA has not promulgated a definition of an AOC unit, an AOC unit is generally an area within a CERCLA investigation site with similar characteristics with respect to contamination and the associated risks to human health and the environment. A CERCLA investigation site may contain one or more AOC units.

**Decontamination fluids:** Any fluids, including aqueous wash water, solvents, and contaminants that are used or generated during decontamination procedures.

**Decontamination solids:** Any solids, including soils and soil cuttings, fill materials, and contaminants that are generated during decontamination procedures.

**Grout:** A fluid mixture of cement and water (neat cement) of a consistency that can be forced through a pipe and placed as required.

**Hazardous waste:** A solid waste that meets the definition of a hazardous waste under RCRA as defined in 40 CFR § 261.3.

**Investigation-derived waste (IDW):** Solid wastes, as defined in 40 CFR § 261.2, directly generated as result of performing the field activities.

**Nonhazardous waste:** A solid waste that does not meet the definition of a hazardous waste as defined in 40 CFR § 261.3 or is excluded from hazardous waste regulation per 40 CFR § 261.4(b).

**Soils and soil cuttings:** Solid material generated from excavation or drilling processes. Soils may include native soils, fill materials, and/or other historical plant waste streams used as fill materials on the site.

**Solid waste:** Any waste stream (solid, liquid or containerized gas) that meets the definition of solid waste under RCRA as defined in 40 CFR § 261.2.

#### 4.0 RESPONSIBILITIES

This section presents a brief definition of the field team roles and responsibilities for management of IDW generated while conducting field investigations. This list is not intended to be a comprehensive list as additional personnel may be involved. Project team member information shall be included in project-specific plans (e.g., work plan, field sampling plan (FSP), quality assurance plan, etc.), and field personnel shall always consult the appropriate documents to determine project-specific roles and responsibilities. In addition, one person may serve in more than one role on any given project.

**Project Manager:** Responsible to ensure that all field team members are properly trained per their responsibilities associated with IDW and that appropriate equipment and facilities are available for appropriate IDW management.

**Field Team Leader (FTL):** Implements the field program and supervises all field team members in the appropriate management of IDW. Ensures that only properly trained personnel are managing IDW on the site.

**Environmental, Health and Safety (EHS) Officer:** Assists the Team Leader in the supervision of all IDW management on site. The EHS officer shall be responsible for all IDW identification

OU3 SOP 12

Rev. No. 0

Date: September 26, 2007

Page 4 of 25

and characterization, on site disposal, off site shipment and disposal, waste accumulation, emergency response and contingency planning, IDW training, and IDW reporting and recordkeeping.

**Project Team Members:** Ensure that they are properly trained prior to any IDW management as well as follow the appropriate IDW procedures and training.

## **5.0 REGULATORY BASIS AND GUIDANCE**

IDW encountered, generated, or managed during the field investigations may contain hazardous substances as defined by CERCLA. Some IDW may be hazardous wastes under RCRA while others may be regulated under other federal laws such as TSCA. These regulatory requirements may be applicable or relevant and appropriate requirements (ARARs) which impact how the IDW is managed.

### **5.1 EPA Guidance on IDW Management**

The management of IDW shall be in accordance with EPA Guidance "Management of Investigation-Derived Wastes During Site Inspections", May 1991 (EPA, 1991). The specific elements of EPA's guidance for IDW management are as follows:

- Characterizing IDW through the use of existing information (manifests, MSDSs, previous test results, knowledge of the waste generation process, and other relevant records) and best professional judgement.
- Delineating an AOC unit for leaving RCRA hazardous soil cuttings within the unit.
- Containerizing and disposing of RCRA hazardous groundwater, decontamination fluids, PPE, and disposable equipment at RCRA Subtitle C facilities.

- Leaving on-site RCRA nonhazardous soil cuttings, groundwater, and decontamination fluids preferably without containerization and testing.

In general, EPA does not recommend removal of wastes from sites, in particular, from those sites where IDW do not pose any immediate threat to human health or the environment. Actions taken during field investigations with respect to IDW that leave conditions essentially unchanged should not require a detailed analysis of ARARs or assurance that conditions at the site after field investigations will comply with the ARARs. At the same time, field personnel ensure that their handling of IDW does not create additional hazards at the site.

In brief, compliance with the NCP can generally be assured by:

- 1) Identifying contaminants, if any, present in the IDW based upon existing information and best professional judgement; testing is not required in most circumstances.
- 2) Determining ARARs and the extent to which it is practicable to comply with them.
- 3) Delineating an AOC unit based upon existing information and visual observation if soil cuttings are RCRA hazardous.
- 4) Burying RCRA hazardous soil cuttings within the AOC unit, so long as no increased hazard to human health and the environment will be created. Containerization and testing are not required.
- 5) Containerizing RCRA hazardous groundwater and other RCRA hazardous IDW such as PPE, disposable sampling equipment, and decontamination fluids for off-site disposal.

## 5.2 Hazardous Waste Regulation

The RCRA hazardous waste regulations are clearly ARARs for hazardous IDW generated and managed during field investigations. However, with the application of EPA IDW guidance, RCRA requirements apply to management of IDW in the following manner:

- If RCRA hazardous IDW is stored or disposed off-site, then comply with all RCRA (and other ARAR) requirements.
- If RCRA hazardous IDW is stored on-site, then comply with RCRA (and other ARAR) requirements to the extent practicable.

For these field investigations, the following general guidance is expected to be practicable and therefore followed, recognizing that each situation will be evaluated against EPA IDW guidance (EPA, 1991) as well as RCRA hazardous waste requirements and other ARARs:

- IDW may be assumed not to be a “listed” hazardous waste under RCRA 40 CFR 261 Subpart D, unless available information about the site suggests otherwise.
- IDW characterization to determine if the IDW exhibits RCRA hazardous waste characteristics do not typically require testing if the characterization can be made by “applying knowledge of the hazardous characteristics in light of the materials or processes used” or by historical testing consistent with 40 CFR § 262.11(c).
- Compliance with the RCRA hazardous waste generator requirements of 40 CFR Part 262 for all RCRA hazardous IDW generated and/or managed (with exception of soil cuttings managed in accordance with the EPA IDW guidance). It is presumed that the RCRA hazardous IDW generated will fall within the large quantity generator (LQG) requirements.
- Land disposal does not occur (and thus the Land Disposal Restrictions [LDR] of 40 CFR Part 268 are not applicable) when IDW soil cutting wastes are:

- Moved, stored or left in place within a single AOC unit;
  - Capped in place;
  - Treated in situ (without moving the IDW to another AOC unit for treatment); or
  - Processed within the AOC unit to improve structural stability (without placing the IDW into another AOC unit for processing).
- Conversely, land disposal does occur (and the LDR of 40 CFR Part 268 are applicable) when IDW soil cutting wastes are:
  - Moved from one AOC unit to another AOC unit for disposal;
  - Moved outside an AOC unit for treatment or storage and returned to the same AOC unit for disposal;
  - Excavated from an AOC unit and placed in a container, tank, surface impoundment, etc. and then re-deposited back into the same AOC.

### 5.3 TSCA PCB Regulation

IDW containing PCBs at detectable levels may be generated, although the concentration of PCBs in any IDW generated is expected to be far below 50 ppm. However, IDW generated will be evaluated for PCBs and managed according to the following per the requirements of 40 CFR Part 761 Subpart D:

- Liquid IDW at concentrations greater than or equal to 50 ppm PCBs will be incinerated off-site at a TSCA-approved incinerator site.
- Nonliquid IDW at concentration greater than or equal to 50 ppm PCBs may be incinerated, treated by an equivalent TSCA-approved method, or disposed in a TSCA chemical landfill off-site.
- IDW at concentrations less than 50 ppm are generally not regulated under TSCA, and may be disposed in an acceptable Subtitle D facility.

## **6.0 DESCRIPTION OF ANTICIPATED IDW MANAGEMENT**

The following subsections provide a description of the anticipated IDW to be encountered, generated, and/or managed at the Libby Asbestos Superfund Site OU3 during field activities and the anticipated management of each. It should be noted that this information is provided for planning purposes, and will be evaluated and may need to be revised based upon actual experience while on site.

### **6.1 Soil and Soil Cuttings**

During field investigations, surface soil samples, samples of mine waste rock, and samples of fine tailings will be collected. Only a small portion of material will be collected for analysis. While the soil and soil cuttings IDW generated will be evaluated on a case-by-case basis, the general approach will follow the EPA guidance for IDW (EPA, 1991) which includes:

- Characterizing the IDW through the use of existing information (previous test results, previous waste characterization, knowledge of the waste generation process, and other relevant records) and best professional judgement.
- Soil and soil cuttings which are not used directly for sample makeup will not be taken outside of the AOC unit in which they were generated.
- Soil and soil cuttings within the AOC where they are generated will be placed back into the same investigation pit, trench, or bore hole and in the same order from which the material was removed, to the extent practicable and unless noted otherwise in the FSP.
- Soil cuttings potentially requiring RCRA disposal will be handled per the procedures presented in Section 7.0 below and disposed in an off-site RCRA facility.

### **6.2 Spent Sampling-Related Equipment**

During field investigations, spent sampling-related equipment will be generated. This may include (but not limited to) plastic sheeting/tarps, rope, bailers, sampling equipment, spent PPE, sample bottles, used containers, packaging materials, and other consumables. Although the vast majority of the spent sampling-related equipment is expected to be nonhazardous, these IDW may contain a listed hazardous waste (e.g., spent solvents) or may exhibit a hazardous waste characteristic (e.g., toxicity from metals).

While the spent sampling-related equipment will be evaluated on a case-by-case basis, the general approach to be followed for spent sampling-related equipment IDW will follow the EPA guidance for IDW (EPA, 1991) which includes:

- Containerizing the spent sampling-related equipment, typically in a satellite accumulation station.
- Characterizing the spent sampling-related equipment IDW through the use of existing information (previous test results, previous waste characterization, knowledge of the contaminants present, and other relevant records) and best professional judgement. This characterization will be documented and maintained as part of the solid/hazardous waste determination records.
- Those spent sampling-related equipment IDW that are determined to be nonhazardous will be disposed of onsite or as municipal waste.
- Those spent sampling-related equipment IDW that are determined to be hazardous will be managed per the procedures presented in Section 7.0 below and disposed in an off-site RCRA facility.

### **6.3 Decontamination Fluids and Solids**

During field investigations, decontamination fluids and solids will be generated. Typically, these will be generated at a common decon area, although there may be more than one decon

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area. Typically, the decontamination IDW will include (but not limited to) washwater from vehicles/equipment, and cleaning agents. Although the vast majority of decontamination IDW is expected to be nonhazardous, this IDW may contain a listed hazardous waste (e.g., spent solvents) or may exhibit a hazardous waste characteristic (e.g., toxicity from metals).

While the decontamination IDW will be evaluated on a case-by-case basis, the general approach to be followed for decontamination IDW will follow the EPA guidance for IDW (EPA, 1991) which includes:

- Containment of decontamination fluids (typically washwater) as generated. The washwater will be segregated from solids to the extent practicable (i.e., solids will be allowed to settle out of the washwater on the decontamination containment pad). Washwater will then be containerized to await waste determination. Solids will also be containerized in a separate container to await waste determination.
- Other decontamination solids such as cleaning utensils and PPE will also be containerized to await waste determination.
- Characterizing the decontamination IDW through the use of existing information (previous test results, previous waste characterization, knowledge of the contaminants present, and other relevant records) and best professional judgement. This characterization will be documented and maintained as part of the solid/hazardous waste determination records.
- The decontamination solids IDW that are determined to be nonhazardous will be disposed of onsite.
- The decontamination liquids IDW that are determined to be nonhazardous will be disposed as a nonhazardous solid waste, preferably on-site.

- The decontamination IDW (either liquid or solid) that are determined to be hazardous will be managed per the procedures presented in Section 7.0 below and disposed in an off-site RCRA facility.

#### **6.4 Drilling, Well Purging, and Development Water**

Generally, water at the Site that is extracted from boreholes, wells or piezometers for the purpose of drilling, development, sampling, or hydraulic testing is considered non-hazardous and will be discharged to designated shallow sumps away from the boreholes or wells at the site. If the water generated is determined to be hazardous will be managed per the procedures presented in Section 7.0 below and disposed in an off-site RCRA facility.

### **7.0 PROCEDURES FOR HAZARDOUS IDW MANAGEMENT**

The following procedures apply to all IDW that have been determined to be hazardous except for soil cuttings IDW that remain with the AOC unit.

#### **7.1 Introduction**

Once an IDW has been determined to be hazardous, the federal RCRA Subtitle C waste management requirements apply to that waste. The scope of this procedure covers the requirements for large quantity generators (LQG) of hazardous IDW which manage the hazardous IDW on site such that RCRA permitting is not required.

#### **7.2 Determine Land Disposal Restrictions**

The 1984 amendments to the RCRA law included a prohibition of land disposal of certain hazardous wastes without first meeting some treatment standards. For the most part, all listed and characteristic hazardous wastes must be treated according to the treatment levels and technologies outlined in 40 CFR Part 268 to reduce the toxicity and/or mobility of hazardous constituents prior to being disposed of on the land, i.e., landfilled. Therefore, a generator must

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determine if the waste is a "restricted waste" under the land ban rules, and if so, off site treatment and disposal is limited. Note that these rules apply only to wastes destined for land disposal which is defined as: placement in or on the land including a landfill, surface impoundment, waste pile, injection well, land treatment facility, salt dome formation, salt bed formation, underground mine or cave, or concrete vault or bunker. Wastes which are shipped off site for disposal other than land disposal are not regulated under the land disposal restriction regulations of 40 CFR Part 268.

Generators of hazardous wastes must determine if the waste is restricted from land disposal under 40 CFR Part 268. The following reporting and recordkeeping requirements apply.

- If a generator determines that he is managing a restricted waste and the waste does not meet the applicable treatment standards, with each shipment of waste, the generator must notify the treatment or storage facility in writing of the appropriate treatment standards;
- If the generator determines that he is managing a restricted waste and the waste can be disposed without further treatment, with each shipment of waste, the generator must submit to the treatment, storage or disposal facility a notice and certification stating that the waste meets the applicable treatment standards;
- If the generator determines that he is managing a waste subject to an exemption from a prohibition on the type of land disposal method utilized for the waste, with each shipment of waste, the generator must submit to the receiving facility a notice stating that the waste is not prohibited from land disposal;
- If the generator is managing prohibited waste in tanks, containers, or containment buildings regulated under 40 CFR 262.34, and is treating such waste in such tanks, containers, or containment buildings to meet applicable treatment standards, the generator must develop a waste analysis plan which describes the procedures the generator will carry out to comply with the treatment standards; and

- If the generator determines whether the waste is restricted based solely on his knowledge of the waste, all supporting data used to make this determination must be retained on-site in the generator's files.

The generator must retain on-site a copy of all notices, certifications, demonstrations, waste analysis data, and other documentation produced pursuant to these requirements for at least three years from the date the waste was last shipped from the site. It should also be noted that it is prohibited to dilute a hazardous waste in order to circumvent the land disposal prohibitions (40 CFR 268.3). Once a waste is determined to be a "restricted waste", an appropriate Treatment, Storage, and Disposal Facility (TSDF) can be selected to properly treat and dispose of the waste.

### **7.3 On-Site Accumulation**

As discussed in Section 5.0 above for each IDW generated, a large quantity generator (LQG) must make the appropriate hazardous waste determination per 40 CFR Part 262.11. If the IDW is determined to be hazardous, then the IDW will typically be stored on-site prior to shipment off-site for disposal. The following requirements apply to all hazardous IDW being stored on-site prior to shipment.

#### **7.3.1 EPA Identification Number (40 CFR Part 262.12)**

Any facility which is a LQG of hazardous wastes must not treat, store, dispose, transport or offer for transportation any hazardous waste without first obtaining a EPA identification number from EPA (or the authorized state). Hazardous wastes cannot be offered to transporters or to treatment, storage or disposal facilities that have not received a EPA identification number.

#### **7.3.2 On-Site Hazardous Waste Accumulation (Storage) (40 CFR 262.34(d))**

Two types of accumulation areas for hazardous waste are permissible for a LQG without RCRA interim status or a Part B permit. These are the "90-day storage area" and the "satellite accumulation station" (SAS). The SAS requirements are discussed below. With regards to a

"90-day storage area", a LQG may store hazardous wastes on-site for up to 90 days or less in a storage area, provided that the following conditions are met:

- If the waste is placed in containers, the requirements of 40 CFR Part 265 Subpart I (container requirements) are met. See below for container requirements;
- If the waste is placed in tanks, the requirements of 40 CFR 265 Subpart J (tank requirements) are met. See below for the tank requirements.
- At closure, the generator closes the storage area per the requirements of 40 CFR 265.111 and 40 CFR 265.114;
- The date which the hazardous waste is placed in the storage area is clearly marked on the container, and the container is clearly marked as "Hazardous Waste";
- The facility complies with 40 CFR Part 265 Subpart C, Preparedness and Prevention (See Section 6.3.3 below);
- The facility complies with 40 CFR Part 265 Subpart D, Contingency Plan and Emergency Procedures (See Section 6.3.4);
- The facility complies with 40 CFR Part 265.16 training requirements (See Section 6.6 below);
- Any hazardous wastes which are stored longer than 90 days must first be granted an extension by EPA (or authorized state).

**90-Day Storage Area Container Requirements (40 CFR Part 265 Subpart I)**

Hazardous waste stored in containers must meet the following requirements:

- Containers must be in good condition, free of leaks;
- Hazardous wastes must be compatible with container (or liner) material;
- Containers must always be kept closed except to add or remove wastes;

- Containers must be handled in a manner to avoid ruptures;
- The storage area must be inspected at least weekly to check for container deterioration; and
- Incompatible wastes must be stored separately with separate secondary containment.

Incompatible wastes are wastes that are unsuitable for co-mingling because the co-mingling could result in any of the following:

- Extreme heat or pressure generation;
- Fire;
- Explosion or violent reaction;
- Formation of substances that have the potential to react violently;
- Formation of toxic dusts, mists, fumes, gases, or other chemicals; and/or
- Volatization of ignitable or toxic chemicals due to heat generation.

#### **90-Day Storage Area Tank Requirements (40 CFR Subpart J)**

LQGs that accumulate or store hazardous wastes in tanks or tank systems must meet the following requirements:

- For tanks existing prior to July 14, 1986, an assessment of tank must be performed and certified by an independent, qualified, licensed engineer. The written certification must be kept on file at the facility (40 CFR 265.191);
- New tank systems (those built after July 14, 1986) must meet tank technical standards and have been certified by an independent, qualified, licensed engineer. The written certification must be kept on file at the facility (40 CFR 265.192);

- New tank systems must have adequate secondary containment and leak detection systems. Existing tanks must be upgraded to meet these standards by the time the tank is 15 years of age (40 CFR 265.193);
- Tanks must be operated to prevent system failure, overflow and spills. Tanks must be operated with sufficient freeboard to prevent overtopping (40 CFR 265.194);
- Inspect the tanks at least once each operating day for the following:
  - Discharge control equipment;
  - Monitoring equipment and controls;
  - Tank level; and
  - Evidence of leaks or spills. (40 CFR 265.195)
- Inspect the tanks at least weekly for corrosion, erosion or leaks;
  - The tank must meet the closure and post-closure care provisions of 40 CFR 265.197; and
  - Store incompatible wastes separately (40 CFR 265.199).

**Satellite Accumulation Station (SAS) Requirements (40 CFR 262.34(c))**

A SAS is a container placed at or near the point of waste generation for the purpose of collecting the waste as it is being generated. For example, a container may be placed in the quality control laboratory for collection of hazardous wastes generated in the laboratory. This SAS may collect up to 55 gallons of hazardous waste or 1 quart of acute hazardous waste. The SAS does not need to meet the requirements of a storage area, provided the following conditions are met:

- The amount of hazardous waste accumulated at the SAS does not exceed 55 gallons (or 1 quart of acute hazardous waste);

- The SAS is located at or near the point of generation where the waste is initially accumulated and is under the control of the operator of the process generating the waste;
- The container used is in good condition, is compatible with the wastes being accumulated, and is kept closed except to add or remove wastes;
- The container is marked with the words "Hazardous Waste" or other words to identify the contents; and
- Once the 55-gallon limit is reached, the date is marked on the container and the container is moved from the SAS within three days to a proper location. For example, the wastes must either be moved to the storage area or be picked up by a waste transporter and moved off-site.

### **7.3.3 Preparedness and Prevention (40 CFR Part 265 Subpart C)**

The following preparedness and prevention steps must be taken concerning the hazardous waste storage area:

- The storage area must be operated and maintained to minimize the possibility of fire, explosions or releases of hazardous waste;
- The facility must have appropriate communication systems, fire-fighting equipment, spill control equipment and decontamination equipment;
- All emergency response systems and equipment must be tested monthly with documentation and maintained to assure proper operation;
- Persons handling hazardous wastes must have immediate access to alarms and/or communication systems;
- The storage area shall have adequate aisle space for emergency response activities; and



- The facility must attempt to make arrangements with the local police, fire departments, emergency response teams, and local hospitals to assure readiness for potential emergencies associated with the storage area.

#### **7.3.4 Contingency Plan and Emergency Procedures (40 CFR Subpart D)**

A LQG that accumulates or stores hazardous waste on site in a 90-day storage area must develop and keep current a contingency plan for the facility. The purpose of the contingency plan is to provide an organized plan of action and delegation of responsibilities and authority to specific facility personnel to respond to emergency situations that may require both the facility and/or outside resources. The contingency plan is designed to minimize hazards to humans or the environment from fires, explosion or any unplanned sudden or non-sudden release of hazardous waste/hazardous waste constituent to air, soil or surface water in compliance with the requirements of 40 CFR 265 Subpart D. A Contingency Plan will be maintained on the site if hazardous IDW are accumulated on-site.

The key components of the contingency plan include the following (40 CFR 265.52):

- A description of the emergency response organization, including designation of the Emergency Coordinator and alternates;
- Response procedures;
- Emergency notification;
- Arrangements with local authorities;
- List of names, addresses and phone numbers of designated emergency personnel and alternates;
- List of emergency response communication equipment and locations;
- Evacuation procedures, routes and alternates; and
- Procedures for amending the plan.

Copies of the plan must be sent to (40 CFR 265.53):

- The Project Manager;
- Lincoln County Sheriff's department;
- Libby fire department; and
- Other agencies as deemed appropriate.

The emergency coordinator (EC) is the key person facilitating emergency preparedness and response. The EC or designated alternate shall be on-site or on-call at all times. The EC and alternates must be trained and thoroughly familiar with the contingency plan, emergency response activities and operation of the facility. The EC must know the locations and characteristics of all waste generated, location of all records within the facility and the facility layout. The EC must have the authority to commit the resources needed to carry out the spill response plan. Any person or department who first discovers any spill of a hazardous waste/material is responsible for notifying the spill response/emergency response coordinator. The EC for the Libby Mine Site field investigations will be the EHS Officer with the Field Team Leader and the Project Manager as alternates.

The contingency plan should be reviewed and immediately amended when:

- Changes in applicable regulations occur;
- The plan fails in an emergency;
- Changes are made to emergency procedures;
- Changes occur in emergency personnel list; or
- Changes occur in emergency equipment list.

#### **7.4 Pre-Transportation Requirements**

Prior to transporting hazardous wastes or offering hazardous wastes for transportation off-site, the generator must comply with the following:

- Package the hazardous wastes in DOT-approved containers per 49 CFR Parts 173, 178 and 179. DOT-approved containers (such as drums) are usually marked as being DOT-approved);
- Label the hazardous wastes according to DOT labeling requirements per 49 CFR Part 172;
- Mark each container (of 110 gallons or less) used in transportation with the following:

HAZARDOUS WASTE - Federal Law Prohibits Improper Disposal. If found, contact the nearest police or public safety authority or the EPA.

- Generator's Name and Address
- Manifest Document Number
- Ensure that the initial transporter placards the transport vehicle with the appropriate placard in accordance with 49 CFR Part 172 Subpart F.

### **7.5 Manifesting Off-Site Shipments of Hazardous IDW**

Any generator which transports or offers for transportation hazardous waste for off-site treatment, storage or disposal must prepare a manifest according to manifest instructions for each shipment of similar hazardous wastes. The manifest must be carefully filled out with each shipment. Take care to follow the instructions and use the terms as listed in the instructions. A generator must designate on the manifest one facility (designated facility) which is permitted to handle the waste described on the manifest (40 CFR 262.20).

The generator must determine if the state to which the wastes are destined (consignment state) requires use of its own manifest. If so, then the consignment state's manifest must be used. If the consignment state does not require use of its manifest, and the state in which the waste

shipment originates (generator state) does, then the manifest from the generator state must be used. If both states have manifests, use the consignment state manifest, making sure that there are sufficient copies to meet the generator state distribution requirements. If neither state requires use of its manifest, then any uniform hazardous waste manifest may be used (40 CFR 262.21).

The manifest must contain at least enough copies such that the generator gets two copies, the transporter gets one copy and the designated facility gets one copy. Some states require additional copies to be sent to the state. At the time of shipment, the generator must keep one copy (the generator copy) of the completed, signed manifest and give the remaining copies to the transporter. Each copy must have the signature of the generator and the transporter at the time of shipment. The original manifest shall be returned to the generator once the shipment reaches the designated facility and the manifest is signed by the designated facility (40 CFR 262.21).

If the original, signed manifest is not received by the generator within a certain number of days, action by the generator is required. These requirements are discussed in the following sections:

- If, after 35 days from the date of shipment, the original manifest copy is not yet received by the LQG, the LQG must contact the transporter and/or the designated disposal facility to determine the status of the hazardous waste (40 CFR 262.42(a)(1)).
- If after 45 days from the date of shipment, the original manifest copy is not yet received by the LQG, the LQG must submit an exception report to the U.S. EPA (or authorized state). The exception report must include a copy of the manifest along with an explanation of efforts to locate the hazardous wastes and the result of these efforts (40 CFR 262.42(a)(2)).

## **7.6 Personnel Training**

Any person, and their immediate supervisor(s), involved in waste management at a LQG facility which stores hazardous waste in a 90-day storage area must undergo initial and annual training

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for hazardous waste management (40 CFR 262.34(a)(4) and 40 CFR 265.16). Facility personnel are required to successfully complete a program of classroom instruction or on-the-job training that teaches them to perform hazardous waste management duties relevant to their jobs. The program must be directed by a person trained in hazardous waste management procedures.

The training must be designed to enable personnel to effectively respond to emergencies by becoming familiar with emergency procedures, emergency equipment and emergency systems, including the following;

- Procedures for using, inspecting, repairing and replacing facility emergency and monitoring equipment;
- Communications or alarm systems;
- Response to fires or explosions; and
- Off-site communication.

Employee training is to be held at regular intervals. Emergency planning information, e.g., the Contingency Plan, also should be provided to state and local emergency response agencies at regular intervals (40 CFR 265.37 and 265.53). Employees required to receive the training cannot work unsupervised until they have completed the training requirements (either classroom or on-the-job training). In addition, facility personnel must take part in an annual review of the initial training.

The following records must be maintained at the facility for employees affected by this training:

- Job title for each position and name of employee filling each job;
- Job descriptions for each position related to hazardous waste management;
- Written description of type and amount of initial and continuing training that will be given to each person filling the various job positions; and

- Documentation that necessary training has been given and completed by each affected personnel.

Training records are required to be kept on current personnel until closure of the facility. For former employees, training records must be kept for at least three years from the date the employee last worked at the facility and may be transferred if the employee stays within the same company (40 CFR 265.16(e)).

## **7.7 Reporting and Recordkeeping**

The following reports are required of a LQG:

- Manifest exception reports as discussed in Section 6.5 above.
- A LQG must submit a Biennial Report to the EPA (or authorized state) every even numbered year by March 1, e.g., March 1, 2008 for the 2007 reporting year. The Biennial Report is to be submitted on EPA form 8700-13A.

The following records are required to be kept for a minimum of three years by the LQG:

- The signed original manifests;
- Biennial reports;
- Exception reports;
- All records pertaining to hazardous waste determinations; and
- Land disposal determination records, notification and certification records.

## **8.0 QUALITY ASSURANCE AND QUALITY CONTROL**

All IDW data must be documented in the field logbooks, field forms, manifests, including rationales deviations from this SOP. The Field Team Leader or designated QA reviewer will

check and verify that IDW documentation has been completed per this procedure and other procedures referenced herein.

## 9.0 REFERENCES

Agency for Toxic Substances and Disease Registry. 2006. Asbestos Exposure and Your Health.

EPA, 1991. Management of Investigation-Derived Wastes During Site Inspections, EPA May 1991, EPA/540/G-91/009

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Libby Superfund Site Operable Unit 3 Standard Operating Procedure

Date: November 20, 2007

SOP TREE-LIBBY-OU3 (Rev. 1)

Title: SAMPLING AND ANALYSIS OF TREE BARK FOR ASBESTOS

**APPROVALS:**

TEAM MEMBER

SIGNATURE/TITLE

DATE

EPA Remedial Project Manager

  
Bonita Lavelle, USEPA RPM

11/20/07

SOP Author

  
William Brattin, SRC

11/20/07

Revision Number	Date	Reason for Revision
0	09/26/2007	--
1	11/20/2007	Modify procedure for sample preparation based on results of pilot-scale laboratory tests

## **1.0 PURPOSE**

The purpose of this Standard Operating Procedure (SOP) is to provide a standardized method for collection and analysis of tree bark samples for asbestos. This procedure will be used by USEPA Region 8 for the Remedial Investigation work for Operable Unit 3 performed at the Libby Asbestos Superfund site.

## **2.0 RESPONSIBILITIES**

The Field Sampling Team Leader is responsible for ensuring that all bark samples are collected in accord with this SOP. The Laboratory Director is responsible for ensuring that bark samples provided to the laboratory for evaluation by this SOP are prepared and analyzed in accord with the requirements of this SOP. It is the responsibility of the Field Sampling Team Leader and the Laboratory Director to communicate the need for any deviations from the SOP with the appropriate USEPA Region 8 Remedial Project Manager or Regional Chemist.

## **3.0 EQUIPMENT**

### **3.1 Field Equipment**

- hole saw (2-inch diameter)
- battery-powered drill
- ½ inch chisel
- flathead screwdriver
- aerosol hair spray
- Ziploc plastic bags
- sample identification tags
- decontamination supplies
- trash bag
- GPS unit
- digital camera
- flagging tape or metal identification tag
- field log book
- field sample data sheet(s) for tree bark
- increment boring device (e.g., Hagloff)
- plastic sheath for age core
- ink pen
- clear packaging tape

### **3.2 Laboratory Equipment /Reagents**

- Disposable Filter funnels with Straight sides. VWR # 145-0020
- Culture Dishes. VWR # 25388-581 (case of 500)
- 47 mm 0.45 micron mixed cellulose ester (MCE) filters
- Glass Petri Dishes

- Glass microscope slides
- Low Temperature Plasma Asher
- Vacuum Evaporator (Carbon Coater)
- Graphite or Carbon rods
- HEPA Laminar Flow Hood
- Acetone Vapor Generator
- Grids
- Fine Forceps
- Grid Clips and Grid Storage Boxes
- Jaffe Wick or Sponge
- Kim wipes or alternative paper
- Transmission Electron Microscope with the following capabilities:
  - 100 Kev
  - fine probe size <250 nm
  - elemental Chemistry via X-Ray Detector
- Large ceramic crucibles (approx. 50 ml capacity or greater)
- Glass stirring rods
- Fumehood
- HEPA filtered Hood
- Ultrasonic Bath producing a rate of energy deposition in the range of 0.08-0.12 MW/m<sup>3</sup>
- Disposable plastic filter funnel apparatus
- Reagent Grade or better Acetone
- Reagent Grade or better HCl

#### 4.0 METHOD SUMMARY

One or more tree bark samples are obtained from selected trees by using a 2-inch hole saw to cut a circular ring in the bark, following by cutting/prying the circular piece of bark from the tree using a sharp chisel. The area to be sampled is sprayed with hair spray prior to sample collection in order to minimize the potential for loss of fibers from the tree bark. In some cases, a core may be obtained from the tree in order to allow verification of the age of the tree.

Tree bark samples are prepared for analysis by high temperature ashing to remove organic matter. The residue is then treated with HCl to dissolve any salts or carbonate component that may be present and applied to a filter which is examined for asbestos using transmission electron microscopy (TEM).

#### 5.0 SAMPLE COLLECTION

Bark samples should be collected from the sampling stations specified in the Sampling and Analysis Plan (SAP). At each specified sampling station, sample collection should be performed as follows:

## 5.1 Select Tree

The species and size of tree selected for sampling should be specified in the project-specific sampling and analysis plan. In the absence of specification, the tree selected for sampling should be a Douglas fir (*Pseudotsuga menziesii*) with a diameter (caliper) of about 8-10 inches. If there are multiple trees that meet these requirements in the vicinity of the sampling station, preference should be given to trees with rough bark, and trees that are in open areas.

## 5.2 Bark Collection

Collect the bark sample from the side of tree facing the mine and from a height of 4-5 feet above the ground.

Steps:

1. Spray the bark collection area with aerosol hairspray and allow to dry.
2. Use a 2-inch diameter hole saw and a battery-powered electric drill to cut a circle in the tree bark. Continue cutting until sawdust changes from red to cream, which indicates that the cambium has been reached (about ½ inch deep).
3. Using a sharp ½-inch metal chisel or flathead screwdriver, cut or pry the circular bark sample off the tree, attempting to maintain the sample in one piece.
4. Place the bark sample in a plastic Ziploc bag.
5. Label the bag with a unique sample identifier.
6. Place clear packaging tape over the sample identifier label.

## 5.3 Tree Age Core Collection

At locations where an age core is to be collected (as specified in the project-specific sampling and analysis plan), collect a core from the tree using a Hagloff manual increment borer or similar device. Place the core in a plastic straw. Crimp and tape the ends of the straw, and label the straw with the same sample identifier as the bark field sample. Place the straw into a Ziploc bag. Label the bag with the same sample identifier as the bark field sample, and place clear packaging tape over the sample identifier label.

## 5.4 Field Documentation

Complete the Tree Bark Field Sample Data Sheet (FSDS) form. Measure and record the diameter of the tree. Obtain and record the GPS coordinates of the tree on the Tree Bark FSDS. Mark the tree with flagging tape or a metal identification tag.

## 5.5 Equipment Decontamination

If dedicated sample equipment is not used, after each sample collection, manually remove any fibrous debris from the hole saw teeth. If resin or pitch is present, use WD40 to clear saw of any residue. Thoroughly clean all collection equipment with isopropyl alcohol wipes. Dry sampling equipment using paper towels. Any spent wipes, paper towels, or other decontamination waste materials must be disposed or stored properly as investigation-derived waste.

## 6.0 SAMPLE PREPARATION AND ANALYSIS

### 6.1 Tree Bark Preparation

#### *Drying and Ashing*

Measure and record the diameter and the thickness of the tree bark sample to an accuracy of  $\pm 2$  mm (about 1/16 of an inch).

Weigh and record the tare weight of a clean crucible. Add the entire tree bark core to the crucible. Place the crucible with bark sample in a drying oven. Heat to 80°C and hold at this temperature until weight stabilizes (at least 6 hours). Record the final weight and calculate the mass of the dried tree bark sample by difference.

Place a lid on the crucible and transfer to a muffle furnace. Ramp up the furnace from a cold start to 450°C. Hold at this temperature for 18 hours or until all organic matter is removed. Allow the crucible to cool. Remove crucible lid and weigh and record the mass of the crucible plus ashed residue. Calculate the mass of the ashed residue by difference.

#### *Acid Treatment*

To the ashed residue, add just enough filtered and deionized (FDI) water (approximately 1-2 mL) to cover the surface of the residue. Slowly add approximately 10-20 mL concentrated HCl to the wetted ash. Typically a visible effervescing is observed. Add the HCl slowly to keep this reaction controlled. A small glass stirring rod is useful at this point to gently stir the ash and expose all material to the acid.

If after 3-5 minutes there is no further visible reaction, proceed to the next step. If bubbling is still occurring, continue observation and gentle stirring for up to an additional 5 minutes.

Dilute the sample by adding FDI water directly to the crucible (approximately 20 mL) using a squirt bottle. Pour the sample into an unused disposable 100 mL specimen container with lid. Rinse out any remaining residue from the crucible into the specimen container. Do not exceed 100 mL total volume. Bring the total volume to 100 mL with FDI water.

Cap the specimen jar and agitate the sample by inversion 5 or 6 times. Loosen the cap slightly and sonicate for 2 minutes. After sonication, tighten the cap and then dry the exterior of the specimen container with kim wipe or equivalent.

#### *Filtration*

Agitate the sample by inversion 5 or 6 times. Withdraw an initial aliquot of 5 to 20 mL of sonicated sample. Transfer this aliquot into a new disposable specimen container with lid. Bring the volume up to approximately 100 mL with FDI water. Cap and agitate by inversion (5 or 6 times).

Filter this entire volume onto a 47 mm mixed cellulose ester (MCE) filter with 0.4 um pore size.

If the filter appears overloaded (overall particulate level > 20%), repeat the process above, selecting a smaller aliquot volume, as suggested by the degree of overloading. Likewise, if the filter looks too lightly loaded, remove and filter a larger aliquot.

Transfer the filter membranes to individual disposable labeled Petri dishes with lids. With the Petri dish covers ajar, dry the filters by air drying.

## 6.2 TEM Examination

Prepare 3 grids for TEM analysis as detailed in International Organization for Standardization (ISO) TEM method 10312, also known as ISO 10312:1995(E). Utilize 2 grids for analysis, and archive 1 grid.

### *Counting rules*

Examine the grids using TEM in accord with ISO 10312, with all relevant Libby site-specific modifications, including utilizing the most recent version of all relevant project specific modifications, including LB-000016, LB-000019, LB-000028, LB-000029, LB-000030, LB-000053, and LB-000066. All fibrous amphibole structures that have appropriate Selective Area Electron Diffraction (SAED) patterns and Energy Dispersive X-Ray Analysis (EDXA) spectra, and having length greater than or equal to 0.5 um and an aspect ratio (length:width)  $\geq 3:1$ , will be recorded on the Libby site-specific laboratory bench sheets. Data recording for chrysotile (if observed) is not required.

### *Stopping rules*

The target analytical sensitivity for sample analysis should be specified in the SAP. In the absence of such specification, the target sensitivity should be no higher than 100,000 cm<sup>-2</sup>. The analytical sensitivity is calculated using the following equation:

$$S = \frac{EFA}{GO \cdot A_{go} \cdot A \cdot F}$$

where:

S	=	Sensitivity (cm <sup>-2</sup> )
EFA	=	Effective filter area (mm <sup>2</sup> )
GO	=	Number of grid openings counted
A <sub>go</sub>	=	Area of one grid opening (mm <sup>2</sup> )
A	=	Area of tree bark sample being analyzed (cm <sup>2</sup> )
F	=	Fraction of original sample deposited on the filter

Count the sample until one of the following occurs:

- The target sensitivity is achieved.
- A total of 50 or more LA structures are observed. In this case, counting may cease after completion of the grid opening that contains the 50<sup>th</sup> LA structure.
- A total of 100 grid openings are counted without reaching the target sensitivity or observing 50 LA structures. In this event, the laboratory should contact EPA asking for direction.

### **6.3 Electronic Data Deliverable**

All data on the number, type and size of LA fibers collected in the laboratory will be provided as an electronic data deliverable (EDD) using the most recent version of the spreadsheet developed for this purpose ("TEM Tree Bark.xls").

### **6.4 Analysis of Core Sample**

The age of the tree will be determined from the core sample in accord with the method of Phipps (1985).

## **7.0 QUALITY ASSURANCE**

### **7.1 Field-Based Quality Assurance**

#### Field Duplicates

Field duplicate tree bark samples will be collected at a frequency specified in the SAP. Each field duplicate should be collected from the same tree at a location no further than 6 inches away from the original bark sample. In the absence of such specification, the rate should be no less than 5%. Field duplicate samples should be labeled with a unique identifier. Sample details should be recorded on the Tree Bark FSDS, including the unique identifier of the "parent" field sample.

#### Equipment Rinsates

If dedicated sampling equipment is not utilized, equipment rinsates should be collected after decontamination of field equipment as described above. The decontaminated equipment (hole saw, chisel) should be rinsed with about 25 mL filtered and deionized water into a glass container. The frequency of rinsate collection should be specified in the SAP. In the absence of such specification, one rinsate sample should be collected per sampling team per day. Equipment rinsate samples should be labeled with a unique identifier. Sample details should be recorded on the Surface Water FSDS.

### **7.2 Laboratory-Based Quality Assurance**

#### Laboratory Blanks

A laboratory blank is a filter that is prepared by processing a clean crucible in the same way that a bark sample is prepared. That is, a clean crucible is placed in the oven (with the sample set) at the

same time that tree-bark samples are undergoing ashing. After ashing, the blank crucible is treated by addition of water and HCl, as described above. The contents of the crucible are then rinsed out, diluted to 100 mL, and an aliquot at least as large as the highest volume aliquot for the sample set is removed and used to prepare a filter for TEM examination. This type of blank is intended to indicate if contamination is occurring at any stage of the sample preparation procedure.

Laboratory blanks should be prepared at a rate specified in the project-specific sampling and analysis plan. In the absence of a project-specific specification, laboratory blanks should be prepared at a rate of 3%.

#### Filtration Blanks

A filtration blank is a clean filter that is prepared by passing 100 mL of laboratory FDI water through it. The purpose of this type of blank is to ensure that the filters are not contaminated in the laboratory, and that fluids used for diluting and processing samples are fiber-free.

Filtration blanks should be prepared at a rate specified in the project-specific sampling and analysis plan. In the absence of a project-specific specification, filtration blanks should be prepared at a rate of 2%.

#### Laboratory Duplicates

Laboratory duplicates will be prepared by applying a second aliquot of ashed residue suspension to a new filter, which is then prepared and analyzed in the same fashion as the original filter. The frequency of laboratory duplicates should be specified in the SAP. In the absence of such specification, the rate should be no less than 5%. Laboratory duplicates should be recorded using the appropriate laboratory quality control field in the TEM EDD spreadsheet.

#### Recounts

The precision of TEM sample results should be evaluated by recounting selected grid openings in accord with the requirements specified in the most recent version of LB-000029.

### **8.0 REFERENCES**

International Organization for Standardization. 1995. Ambient Air – Determination of asbestos fibres – Direct-transfer transmission electron microscopy method. ISO 10312:1995(E).

Phipps, R.L. 1985. Collecting, Preparing, Cross-dating and Measuring Tree Increment Cores. U.S. Geological Survey Water Resources Investigations Report 85-4148

Ward TJ, T Spear, J Hart, C Noonan, A Holman, M Getman, and JS Webber. 2006. Trees as Reservoirs for Amphibole Fibers in Libby, Montana. Science of the Total Environment 367: 460-465.



Libby Superfund Site Operable Unit 3 Standard Operating Procedure

Date: June 14, 2010

SOP ABS-LIBBY-OU3 (Rev. 0)

Title: ACTIVITY-BASED SAMPLING FOR ASBESTOS

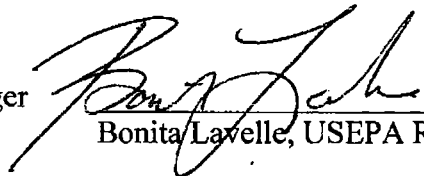
**APPROVALS:**

TEAM MEMBER

SIGNATURE/TITLE

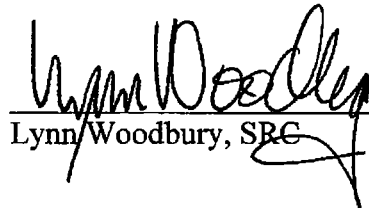
DATE

EPA Remedial Project Manager

  
Bonita Lavelle, USEPA RPM

6/14/10

SOP Author

  
Lynn Woodbury, SRC

6/14/10

Revision No.	Date	Reason for Revision
0	06/14/2010	--

## 1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide a standardized method for collection of air samples during activity-based sampling (ABS) for Libby amphibole asbestos (LA). This SOP was developed based on procedures provided in ERT SOP #2084 (Rev 0) *Activity-Based Sampling for Asbestos* and Libby SOP EPA-LIBBY-01 (Rev 1) *Sampling for Asbestos Fibers in Air*.

This procedure will be used by USEPA Region 8 for the Remedial Investigation work for Operable Unit 3 (OU3) performed at the Libby Asbestos Superfund site.

## 2.0 RESPONSIBILITIES

All staff involved with the collection of ABS air samples are responsible for understanding and implementing the requirements contained herein as well as other related project-specific requirements.

Team Leader – The team leader is responsible for communication with EPA regarding status and progress of the sampling event and providing support to field team staff to ensure all necessary resources are available for implementation of the ABS program.

Field Team Leader (FTL) – The FTL is responsible for ensuring that the specifics related to the ABS program described in this procedure are followed by all staff and that all quality assurance/quality control (QA/QC) procedures related to this program are implemented.

Field Team Members – The field team members are responsible collection and documentation of samples as described in the applicable Sampling and Analysis Plan (SAP) and this SOP.

## 3.0 METHOD SUMMARY

At present, models are not available to accurately predict asbestos exposure in air that result from disturbance of an LA-contaminated source based only on measures of LA in the source (e.g., soil). Therefore, personal monitoring in the form of ABS is the most appropriate technique to estimate exposure (USEPA 2008). As part of ABS, USEPA or contractor personnel trained in hazard recognition and mitigation, serve as surrogates for the potentially exposed populace of interest. ABS simulates particular activities in order to mimic and evaluate or predict personal exposures from disturbance of materials potentially contaminated with asbestos. Air is drawn through a fine-pore filter in order to trap any suspended particulate material in the air, including suspended asbestos fibers. ABS air samples are collected from the breathing zone of the subject at an appropriate air flow rate. These filters are then examined using an appropriate microscopic technique to observe, characterize, and quantify the number of asbestos fibers on the filter. Air filters collected in this way are suitable for examination by a variety of microscopic techniques, including transmission electron microscopy (TEM), phase contrast microscopy (PCM), and scanning electron microscopy (SEM).

#### 4.0 FIELD EQUIPMENT

- Personal sampling pump – The selected sampling pump will be capable of a flow-rate and pumping times sufficient to achieve the desired air sample volume. The sampling pump will provide a non-fluctuating air-flow through the filter, and will maintain the initial volume flow-rate to within  $\pm 10\%$  throughout the sampling period. For personal air sampling, a portable high volume AC powered sampler or low volume battery-operated pump are acceptable, depending on whether the activities of the individual are impaired by the tethering imposed by the power cord needed for the high volume pump.
- Filter cassettes – A commercially available, 25-millimeter (mm), three-piece cassette with a 50 mm electrically-conductive extension cowl pre-loaded with a 0.8 micrometer ( $\mu\text{m}$ ) mixed cellulous ester (MCE) filter.
- Inert tubing – Tygon tubing used in the sampling train to connect the outflow end of the sample cassette to the sampling pump. Tubing has a 1/4" inner diameter and 7/16" outer diameter.
- Rotameter – A rotameter calibrated such that the operator can measure flow rates to  $\pm 5\%$  accuracy at the expected sampling flow rate.
- Rotameter calibration source – A bubble buret or other primary calibration standard may be used to calibrate the rotameter.
- Field sample data sheets (FSDS) – Specific data related to the collection of each sample will be recorded on an OU3-specific ABS air FSDS (see Attachment 1). This sheet will contain all relevant information regarding equipment used, flow rates, and collection times.
- Chain of custody (COC) forms – Documentation of chain of custody signatures/dates and requested analyses for each sample collected will be recorded on an OU3-specific COC form.
- Field logbook
- Spring clips
- Plastic bags
- Sample labels
- Clear packaging tape
- Ink pen
- Personal protective equipment (PPE)

- Power sources (e.g., batteries, power inverters, generators)
- Miscellaneous tools (e.g., screwdriver, pliers, scissors)

## **5.0 REAGENTS**

Reagents are not required for the collection or preservation of asbestos air samples.

## **6.0 SAMPLING PROCEDURES**

### **6.1 Identification of ABS Areas**

When selecting areas for ABS, consideration should be given to the potential for off-site migration of contaminants and possible exposure of the public. Within the constraints of ABS, to the degree practical, particulate generation migration off-site should be minimized, and constraints or mitigation protocols established to eliminate public exposure.

### **6.2 Selection of Air Volumes and Flow Rates**

Target air volumes and flow rates will be specified in the applicable SAP. For activities that generate a large quantity of dust, flow rates and sample times may be adjusted to ensure the sample filter has proper loading for the required analytical analysis and sensitivity goals.

While high flow rate are desirable because they increase the volume of air sampled, high flow rates may result in filter damage due to failure of its physical support associated with increased pressure drop, leakage of air around the filter mount so that the filter is bypassed or damage to the asbestos structures (e.g., breakup of bundles and clusters) due to increased impact velocities (ISO 10312). High flow rates can also tear the filters during initial pump startup due to the shock load placed on the filter when the pump is first started.

In no event shall a sample be collected at a flow rate lower than 0.92 L/min, since the linear flow velocity would fall below 4 cm/sec, which is the minimum velocity specified by ISO 10312.

### **6.3 Sample Collection**

There are a variety of ABS activities that can be performed (e.g., ATV riding, raking, jogging). The specific ABS activity to be performed will be specified in the appropriate SAP. The SAP should include a detailed ABS "script" of these activities.

For all ABS events, except as noted otherwise, air samples will be collected from the breathing zone of the event participants. The breathing zone can be visualized as a hemisphere approximately 6 to 9 inches around an individual's face. Breathing zone samples provide the best approximation of the concentration of contaminants in the air that an individual is actually breathing. Specific breathing zone heights should be determined on a project-by-project basis.

## Libby Superfund Site Operable Unit 3 Standard Operating Procedure

ABS air samples should be collected as follows:

1. Place a sample label on the sample cassette indicating the unique Index ID.
2. Secure the cassette on the lapel just below the face of the individual being monitored using spring clips. Orient the cassette so the open face of the cowl is pointing downward to avoid any particles entering the filter by precipitation.
3. Remove the protective cap over the open face of the cowl and turn on the calibrated pump. Store cassette covers and end plugs in a clean area (e.g., closed bag) during the sampling period).
4. Record the starting time, initial flow rate, and all other relevant sample information on the appropriate FSDS form.
5. Perform the ABS activity in accordance with the script specified in the appropriate SAP.
6. For sampling events that last longer than 2 hours, perform a check of the filter cassette and flow rate every 2 hours. During this check,
  - inspect the filter cassette to ensure that it has not been disturbed
  - inspect the filter for overloading and particle deposition (note: if particle deposition is observed on the inside of the cowl, it may be necessary to ground the cowl to reduce static charge)
  - check the pump flow rate (see Section 7.3 below)
7. After the sampling period has elapsed, measure the end time and ending flow rate on the appropriate FSDS form.
8. Turn off the pump and remove the cassette from the pump.
9. Attach and secure a sample seal around each sample cassette in such a way as to assure that the end cap and base plug cannot be removed. Tape the end of the seal together and initial/date the seal.
10. Place each sample cassette in a plastic sample bag. Each bag should be marked with the same Index ID as the sample cassette. Place clear packaging tape over the sample identification label.

If it is necessary to relieve a participant from the activity, another sample collector should be suited and ready to participate in the ABS prior to the personnel exchange. The participant will stop the activity, remove any sampling apparatus, and pass it to the relief participant. The original participant will assist the relief participant with donning the sampling apparatus. If the exchange is anticipated to take less than 60 seconds, the sampling pumps and event time clock will not be halted during the exchange. If the exchange requires more than 60 seconds, the pump and event clock will be stopped until activity is reinitiated.

#### 6.4 Sample Handling and Storage

Place a sample label on the cassette indicating a unique sampling number. Do not put sampling cassettes in shirt or coat pockets as the filter can pick up fibers or a static charge that could disturb the dust deposited on the filter media.

Samples must be handled gently with the filter inlet facing upward to avoid disturbing the particulate deposited on the filter and to minimize the potential of imparting a static charge to the cassette, which might alter the particulate deposition on the filter media.

To the best extent possible, the sampling cassettes should be placed right side up so that the cassette inlet cap is on top and cassette base is on bottom. Place samples into a shipping container and use enough packing material to prevent jostling or damage. Samples must be handled gently so as not to disturb the dust deposited on the filter media. Do not use vermiculite or any other type of fibrous packing material for samples.

Ship the cassettes to the analytical laboratory under COC procedures.

### 7.0 CALIBRATION PROCEDURES

Each sampling pump will be calibrated before the start of each air sample collection event. This is to ensure that each sampling pump is measuring the flow rate or volume of air correctly.

#### 7.1 Calibration of Rotameter with an Electronic Calibrator

Rotameters used for pump calibration are calibrated to a primary flow standard. Calibration of the rotameter to the primary flow standard shall occur once at the beginning of the sampling program, and quarterly thereafter as needed. Procedures for rotameter calibration with the primary flow standard meter are as follows:

1. If the electronic calibrator does not automatically adjust to account for temperature and pressure changes, obtain the actual temperature and pressure in Libby, MT from the local National Oceanic and Atmospheric Administration (NOAA) weather station or from temperature and weather reference centers. Record actual temperature and pressure in the fields provided on the Precision Rotameter Calibration Data Sheet (Attachment 2).
2. Set up the calibration train as shown in EPA SOP #2015 Figure 4 (Attachment 3) with the sampling pump, rotameter, and primary flow meter.
3. Hold the rotameter as vertical as possible.
4. Turn the primary flow standard and sampling pump on.
5. Adjust the pump until the desired flow rate is attained.
6. Calibrate rotameter to desired ball reading, as read from the middle of the flow ball. Record value in the Ball Reading column on the rotameter calibration data sheet.
7. Check adjusted flow rate of sample pump to the primary flow standard. Ten repetitive flow measurements will be averaged and that result recorded in the flow rate column for the selected interval.

8. Repeat this process at 10 intervals over the range of the precision rotameter.
9. Input data into rotameter calculation sheet to generate the corrected flow rate.

## 7.2 Calibration of Sampling Pump with a Rotameter

Prior to the start of each air sample collection event, each sampling pump will be calibrated with a rotameter that has been calibrated as described above. The procedures used for sampling pump calibration are as follows:

1. Set up the calibration chain as shown in EPA SOP #2015, Figure 5 (Attachment 3) using a rotameter, sampling pump and a representative sample cassette. The sample cassette to be used for sampling is installed between the pump and the calibrator.
2. To set up the calibration train, attach one end of tubing to the sample cassette base; attach the other end of the tubing to the inlet plug on the pump. Another piece of tubing is attached from the sample cassette cap to the rotameter.
3. Hold the rotameter as vertical as possible.
4. Turn the sampling pump on.
5. Adjust the sampling pump until the middle of the float ball on the rotameter is lined up with the pre-calibrated flow rate value.
6. Each rotameter used for field calibration will be transported to and from each sampling location in a sealed zip-top plastic bag. The sample cassette cap and attached tubing for rotameter checks will be stored in the site enclosure in a plastic bag.

## 7.3 Adjustment of Flow Rates During Flow Checks

Flow checks will be performed every 2 hours during the ABS activity. During these checks, flow rates will be adjusted back to the target rate. Adjustment of flow rates during flow checks will be performed as described below. Should the flow rate change from the target flow rate, the following procedure will be used to make the adjustment:

1. Connect the rotameter as described in Section 7.2 steps 1 to 3.
2. Record the observed flow rate and time of observation.
3. Adjust the flow rate to the target flow.

Attachment 4 illustrates the volume tracking spreadsheet that will be used in the field to determine the time required for sample collection. Copies of all volume tracking spreadsheets will be provided to the project data manager at the conclusion of each sampling event. Electronic copies are suitable and will be placed in the project-specific FTP site within one week from completion of each sampling event.

## 8.0 CALCULATION OF AIR VOLUME

Air volume for a sample is calculated based on the flow rate and sample collection duration as follows:

$$\text{Air Volume (L)} = \text{Flow Rate (L/min)} \times \text{Sample Collection Duration (min)}$$

The sample volume should be recorded on the COC form which is provided to the analytical laboratory. For lot blanks and field blanks, the associated air volume should be recorded as zero.

If the flow rate changes by more than 5% during the sampling period, the average of the pre- and post-sampling rates will be used to calculate the total sample volume. If at any time the measurement indicates that the flow-rate has decreased by more than 30%, sampling should be terminated. Depending on the type of sampling pump used, it may be possible to salvage the sample if sufficient volume was collected; however, it may not be representative of the time it takes for the actual activity to be completed. Depending on the type of sampling pump used, the actual sampling time in hours and minutes before the sampling fault may be displayed and an actual sample volume calculated. If the fault was due to battery failure, it may be possible to check the post-sampling flow.

## 9.0 QUALITY ASSURANCE / QUALITY CONTROL

### Lot Blanks

Before sampling begins, two cassettes from each filter lot should be randomly selected and submitted for analysis. These lot blanks will be analyzed for asbestos by the same method as used for the field samples to ensure that cassettes are free from asbestos contamination. The area of the filter examined should be no less than 0.1 mm<sup>2</sup>, unless specified otherwise in a project specific plan. If asbestos is detected in any lot blank, the entire cassette lot should be rejected for use.

### Field Blanks

Field blanks are used to determine if any contamination has occurred during sample handling. A field blank is prepared by opening the filter cassette in the area where the field samples will be taken, and then closing and sealing the sample in a manner similar to the field samples. Field blanks should be assigned a unique Index ID. Field blanks will be collected at a frequency specified in the project-specific SAP. In the absence of such specification, the rate should be no less than 5%. The area of the filter examined should be no less than 0.1 mm<sup>2</sup>, unless specified otherwise in a project specific plan.

### Field Duplicates

Field duplicates are used to evaluate the sampling and analysis variability across air samples. Field duplicate air samples will be collected at a frequency specified in the project-specific SAP. In the absence of such specification, the rate should be no less than 5%. Each field duplicate will be a sample collected concomitantly with the field sample. Field duplicate samples should be labeled with a unique Index ID. Sample details should be recorded on the appropriate FSDS form, including the unique Index ID of the "parent" field sample.

## 10.0 HEALTH AND SAFETY



## Libby Superfund Site Operable Unit 3 Standard Operating Procedure

Inhalation of asbestos fibers is hazardous to human health. When working with potentially hazardous materials, personnel should follow USEPA, OSHA, and corporate health and safety procedures. For all ABS, appropriate PPE, including Tyvek coveralls, protective gloves and foot wear, and a respirator with HEPA filter cartridges (P-100 or equivalent) should be worn to protect participants. Details regarding PPE and other protective measures should be specified in the site-specific Health and Safety Plan (HASP). Special consideration should be given to the physical safety of the event participants as well as heat stress associated with performing vigorous activities in impermeable clothing.

### 11.0 REFERENCES

International Organization for Standardization. 1995. Ambient Air – Determination of asbestos fibres – Direct-transfer transmission electron microscopy method. ISO 10312:1995(E).

USEPA. 2008. Framework for Investigating Asbestos-Contaminated Sites. Report prepared by the Asbestos Committee of the Technical Review Workgroup of the Office of Solid Waste and Emergency Response, U.S. Environmental protection Agency. OSWER Directive #9200.0-68. [http://epa.gov/superfund/health/contaminants/asbestos/pdfs/framework\\_asbestos\\_guidance.pdf](http://epa.gov/superfund/health/contaminants/asbestos/pdfs/framework_asbestos_guidance.pdf)

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ATTACHMENT 1

FIELD SAMPLE DATA SHEET (FSDS) FOR ABS AIR SAMPLE COLLECTION

# LIBBY OU3 FIELD SAMPLE DATA SHEET (FSDS) (rev 0)

## PHASE IV-A ACTIVITY BASED SAMPLING (ABS)

Field Logbook No \_\_\_\_\_

Page Numbers \_\_\_\_\_

Location Identification \_\_\_\_\_

Sampling Team \_\_\_\_\_

Sampler Initials \_\_\_\_\_

GPS ID \_\_\_\_\_

Data Item		Cassette 1		Cassette 2		Cassette 3	
Index ID		AFFIX LABEL HERE		AFFIX LABEL HERE		AFFIX LABEL HERE	
Person (circle)		1	2	1	2	1	2
Scenario (see key below)							
Pump (circle)		HV	LV	HV	LV	HV	LV
Sample Type (circle)		Personal	Stationary	Personal	Stationary	Personal	Stationary
Field QC Type (circle)		FS - Field Sample FB - Field Blank Lot Blank		FS - Field Sample FB - Field Blank Lot Blank		FS - Field Sample FB - Field Blank Lot Blank	
Pump ID Number							
Rotameter ID Number							
		Start	Stop	Start	Stop	Start	Stop
Date (mm/dd/yy)							
First	Time (hh:mm)						
	Flow (L/min)						
Second	Time (hh:mm)						
	Flow (L/min)						
Total Volume (L)							
Pump fault? (circle)		Yes	No	Yes	No	Yes	No
Field Comments							
Cassette Lot Number:							
Entered By (Provide initials):				Validated By (Provide initials):			

**ABS Scenario Key:**

Population	ABS Scenario	ABS Script	Population	ABS Scenario	ABS Script
Recreational visitor along Rainy Creek	Hiking along Rainy Creek	1	USFS Firefighter (ground-based)	Cutting firelines by hand	3d
Residential wood harvester	Driving to and from harvest area	2a		Cutting firelines with heavy equipment	3e
	Cutting and hauling firewood	2b		Personal monitors worn during simulated wildfire	4a
USFS Worker (forest management activities)	Trail maintenance	3a		Stationary monitors activated during simulated wildfire burns	4b
	Thinning trees	3b	USFS Firefighter (pilot of aircraft)	Fly through smoke from simulated wildfires	5a
	Stand exam	3c		Fly through smoke from authentic wildfires	5b

For Data Entry Completion (Provide Initials)	Completed by:	QC by:
----------------------------------------------	---------------	--------

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Rev. No. 0

June 14, 2010

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## ATTACHMENT 2

### PRECISION ROTAMETER CALIBRATION DATA SHEET

Calibration Date: \_\_\_\_\_ Calibration By: \_\_\_\_\_  
Rotameter ID: \_\_\_\_\_ Primary Standard ID: \_\_\_\_\_

If calibrator does not automatically adjust for temperature and pressure, specify –

Actual Temp (°F): \_\_\_\_\_ Actual Pressure (in. Hg): \_\_\_\_\_

Ball Reading = Y (mid-ball)

Flow Rate =  $X_1$  (L/min)

1. \_\_\_\_\_  
2. \_\_\_\_\_  
3. \_\_\_\_\_  
4. \_\_\_\_\_  
5. \_\_\_\_\_  
6. \_\_\_\_\_  
7. \_\_\_\_\_  
8. \_\_\_\_\_  
9. \_\_\_\_\_  
10. \_\_\_\_\_

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#### Rotameter Calibration Procedure:

1. Obtain the actual temperature and actual pressure in Libby, MT from the MET station. If calibrator does not automatically adjust for temperature and pressure, record the actual temperature and actual pressure in the fields provided above.
2. Calibrated rotameter to desired ball reading with a sampling pump and cassette in-line. Cassette must be the same type and from the same lot of cassettes that will be used for sampling. Record value in Ball Reading column.
3. Check adjusted flow rate of sample pump to the Dry Cal flow calibrator primary flow standard. 10 repetitive flow measurements will be averaged and that result recorded in the Flow Rate column for the selected interval.
4. Repeat this process at 10 intervals over the range of the precision rotameter.
5. Input data into rotameter calculation sheet to generate the corrected flow rate.

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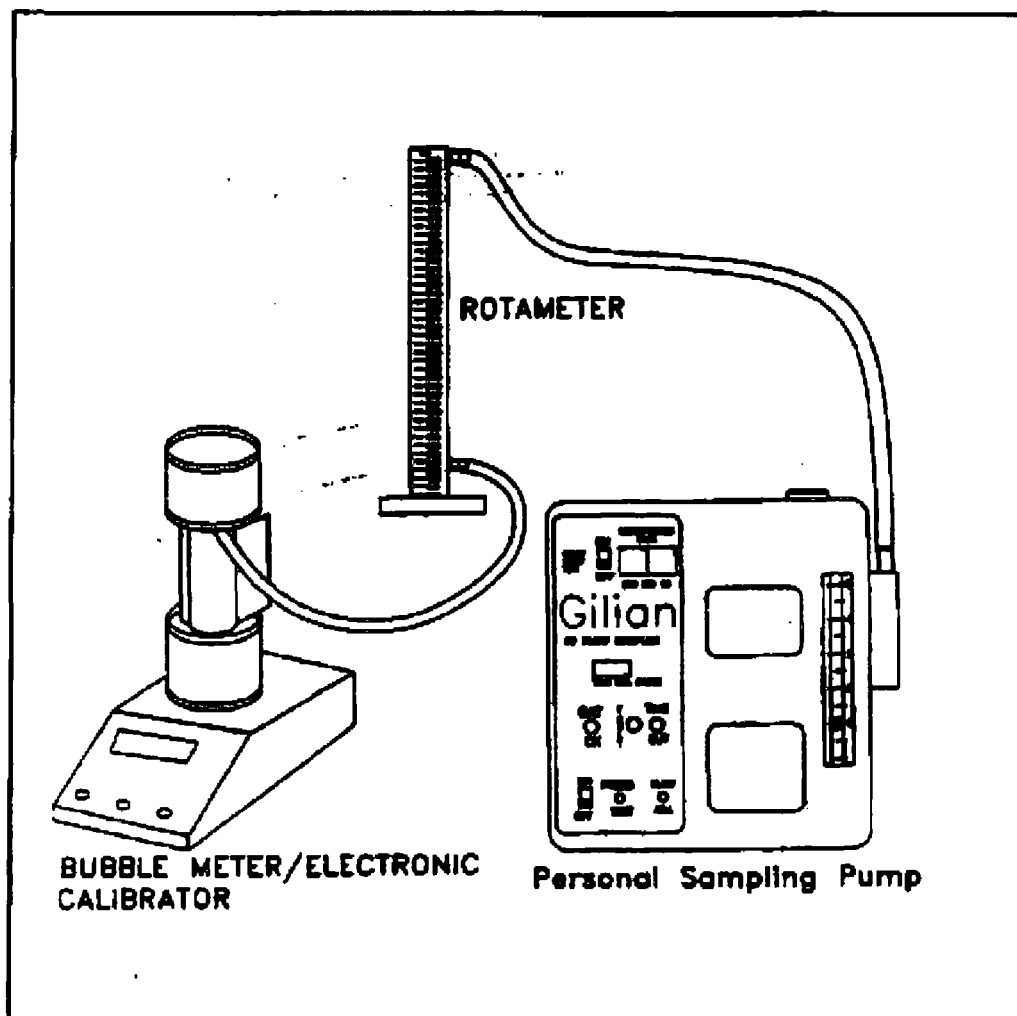
ATTACHMENT 3

CALIBRATION TRAIN FIGURES  
(from EPA SOP #2015)

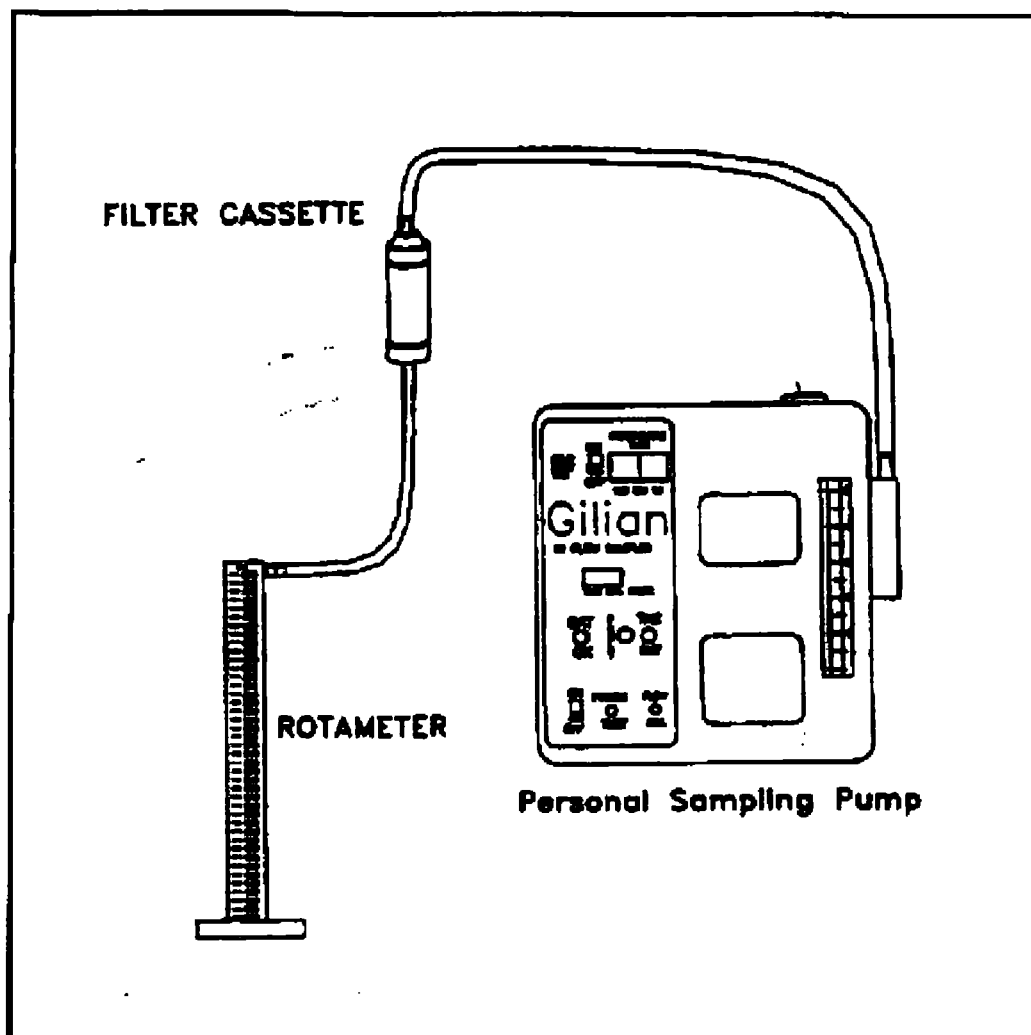
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FIGURE 4. Calibrating a Rotameter with a Bubble Meter



**FIGURE 5. Calibrating a Sampling Pump with a Rotameter**



\_\_\_\_\_

[illegible]

**ATTACHMENT C**

**LIBBY LABORATORY MODIFICATIONS FOR TEM ANALYSES**

**LB-000016**

**LB-000019**

**LB-000028**

**LB-000029b**

**LB-000030**

**LB-000066c**

**LB-000085**

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**Request for Modification**  
To  
**Laboratory Activities**  
LB-000016

**Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.**  
**File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:**

All Lab Applicable forms – copies to: EPA, Volpe, CDM-Denver, All project labs

Individual Lab Applicable forms – copies to: EPA, Volpe, CDM-Denver, Initiating Lab

Method (circle one/those applicable): TEM-AHERA, TEM-ISO 10312, PCM-NIOSH 7400, PLM-NIOSH 9002,  
EPA/600/R-93/116, ASTM D5755-95, EPA/540/2-90/005a, Other: \_\_\_\_\_

Requester: Jeanne Orr Title: President  
Company: Reservoirs Environmental, Inc. Date: December 2, 2002

**Description of Modification:**

Permanent modifications and clarifications to the Transmission Electron Microscopy analysis of air samples using ISO 10312. The purpose of the attached is to document permanent historic modifications & clarifications.

**Reason for Modification:**

To optimize the efficiency of air sample analysis and to provide consistency in analytical procedures and data recording in the project laboratories.

**Potential Implications of this Modification:**

Modifications reflect changes necessary to clarify ISO requirements in relation to project-specific issues. No negative implications to these modifications are anticipated. Positive implications are consistency in procedures between and within project laboratories and documentation of those procedures.

Laboratory Applicability (circle one): ☒ All Individual(s) \_\_\_\_\_

**Duration of Modification (circle one):**

Temporary Date(s): \_\_\_\_\_

Analytical Batch ID: \_\_\_\_\_

Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

☒ Permanent (complete Proposed Modification Section) Effective Date: HISTORIC

Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by TEM analysts.

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):

Please see the attached for the description of the TEM-ISO clarifications/modifications

Technical Review: R.K. Mahony E.M.S. Date: 23 April 2003  
(Laboratory Manager or designate)

Project Review and Approval: [Signature] Date: 4 April 2003  
(Volpe: Mark Raney)

Approved By: Mary G. Gade Title: Project Chemist Date: 3 April 2003  
(USEPA: Mary Gade)

**1. Modification:**

The ISO method requirement is if the specimen grid exhibits more than approximately 10% obscuration on the majority of the grid openings, the specimen shall be designated as overloaded. A rejection criteria of >25% obscuration and <50% intact grid openings will be used for this project. The 25 % overload criteria resulted from various communications that took place 29 December 1999 between EPA Region 8, Camp Dresser McKee, Volpe Center, and Reservoirs.

**2. Modification:**

ISO 10312 is a direct preparation method. If samples are visibly overloaded or contain loose debris and they have not been previously analyzed (the filter is whole) they will be prepared indirectly according to procedures described in ASTM D5755-95. If the sample has been previously analyzed or rejected in the microscope (section removed from the filter), prepare the sample indirectly according to EPA/540/2-90/005a by plasma ashing a portion of the original filter and depositing an aliquot on a secondary filter. Secondary filters will be analyzed according to the ISO counting rules for this project. Calculations are modified to contain a dilution factor. This indirect preparation procedure is embraced to enable the capture of data from samples that otherwise would be rejected.

**3. Clarification:**

Stopping rules for ISO analyses are completion of the grid opening on which the 100<sup>th</sup> asbestos structure has been recorded, or a minimum of four grid openings. For this project, a maximum of ten grid openings will be read unless specifically instructed otherwise.

If abundant chrysotile is present, the chrysotile count may be terminated at the end of the grid opening where the 100<sup>th</sup> chrysotile structure is counted. The analysis will continue recording amphibole fibers only until the remaining grid openings to be analyzed are completed. The grid opening location designation will be followed by a "\*" to indicate the grid openings where only amphibole asbestos was recorded, i.e. K6\*.

This clarification in structure counting and recording is to provide consistency in analytical procedures and data recording in the project laboratories.

**4. Modifications and clarifications: Structure counting and recording**

- a. **Modification:** Non-asbestos structures are not being recorded. This project-specific modification stems from our need only to quantify contaminants of concern: the asbestos levels at a given sample location.
- b. **Modification:** The overall dimensions of disperse clusters (CD) and disperse matrices (MD) will not be recorded in two perpendicular directions. The matrix type and individual structures associated with the matrix or cluster will be recorded as described in the ISO method.
- c. **Modification:** Structures that intersect a non-countable grid bar will be recorded on the count sheet but excluded from the structure density and concentration calculations.
- d. **Modification:** If a structure originates in one grid opening and extends into an adjacent grid opening, providing that it does not intersect a non-counting grid bar, the entire length of the fiber is recorded.
- e. **Clarification:** If a structure intersects both a countable and a non-countable grid bar, the observed length of the structure will be recorded.

These modifications and clarifications in structure counting and recording are to provide consistency in analytical procedures and data recording in the project laboratories.

**Mahoney, Ron**

---

**From:** Raney, Mark [RANEY@VOLPE.DOT.GOV]  
**Sent:** Tuesday, April 22, 2003 11:09 AM  
**To:** 'Mahoney, Ron'  
**Subject:** FW: VOLPE Approved MODS: LB-000015, LB-000016, and LB-000017



LB-000015\_rev (MR)  
4-4-03 email...



LB-000016\_rev (MR)  
4-4-03 email...



LB-000017\_rev (MR)  
4-4-03 email...

FYI

> -----Original Message-----

> From: Raney, Mark  
> Sent: Friday, April 04, 2003 9:31 AM  
> To: 'Beckham, Richard'; 'Goldade.mary@EPAMail.epa.gov'; 'mgoldade@peakpeak.com'  
> Cc: Autio, Anni  
> Subject: VOLPE Approved MODS: LB-000015, LB-000016, and LB-000017

> Volpe provides approval to revised MODs LB-000015, LB-000016, & LB-000017 as attached. The attached MODs include the following changes to the previous versions (received 4/1/03).

> \* The date indicated in the "Effective Date" field was removed and replaced with "HISTORIC"  
> \* Under the "Description of Modification" section the following sentence was added "The purpose of the attached is to document permanent historic modifications & clarifications."

> If you have any questions as to these changes or the reason behind them let me know. Please proceed with distribution of the accepted versions of the attached for final hardcopy signature.

> Mark.

> > <<LB-000015\_rev (MR 4-4-03 email).doc>>> > <<LB-000016\_rev (MR 4-4-03 email).doc>>> > <<LB-000017\_rev (MR 4-4-03 email).doc>>

> -----Original Message-----

> From: Beckham, Richard [mailto:BeckhamRE@cdm.com]  
> Sent: Tuesday, April 01, 2003 10:47 AM  
> To: 'Goldade.mary@EPAMail.epa.gov'; 'RANEY@VOLPE.DOT.GOV'; 'mgoldade@peakpeak.com'  
> Cc: Autio, Anni  
> Subject: FW: LB-000015, LB-000016, and LB-000017

> For your review and approval.

> - Richard Beckham

> -----Original Message-----

> From: Mahoney, Ron [mailto:Rmahoney@EMSL.com]  
> Sent: Monday, March 31, 2003 6:11 PM  
> To: Beckham, Richard  
> Subject: LB-000015, LB-000016, and LB-000017

> Richard,

> These should be final. The only recent revision is the addition of the  
> Effective Date. These need to go to Mark and Mary for their final blessing.



> <<LB-000015(rev 3\_31\_03).doc>> <<LB-000016 rev. (3\_31\_03).doc>>  
> <<LB-000017 rev(3\_31\_03).doc>>

>

> R.K. Mahoney  
> Senior Analyst  
> Special Projects Coordinator  
> EMSL Analytical, Inc.  
> Westmont, NJ  
> 800.220.3675, x1218  
> rmahoney@emsl.com

>

> << File: LB-000015(rev 3\_31\_03).doc >> << File: LB-000016 rev. (3\_31\_03).doc >> << File: LB-000017 rev(3\_31\_03).doc >>

**Mahoney, Ron**

---

**From:** Raney, Mark [RANEY@VOLPE.DOT.GOV]  
**Sent:** Wednesday, April 23, 2003 9:02 AM  
**To:** 'Mahoney, Ron'  
**Subject:** FW: EPA APPROVED CONDITIONAL: LB-000015, LB-000016, and LB-000017



LB-000015 (rev  
3\_31\_03).doc



LB-000016 rev.  
(2\_31\_03).doc



LB-000017  
rev(3\_31\_03).doc

Ron,

I almost forgot to forward you this....

See Mary's earlier email below, regarding EPA's approval for MODs LB-15, 16, & 17.

Let me know if you have any questions.

Mark.

-----Original Message-----

**From:** Goldade, Mary [mailto:Goldade.Mary@epamail.epa.gov]  
**Sent:** Thursday, April 03, 2003 5:49 PM  
**To:** Beckham, Richard  
**Cc:** Autio, Anni; 'mgoldade@peakpeak.com'; 'RANEY@VOLPE.DOT.GOV'  
**Subject:** EPA APPROVED CONDITIONAL: LB-000015, LB-000016, and LB-000017

Richard,  
Mark will modify LB-000015, 16 & 17 to indicate that the Effective Date  
is: Historical.  
EPA approves these mods with this changed completed.

"Beckham,  
Richard" To: Mary Goldade/EPR/R8/USEPA/US@EPA, "RANEY@VOLPE.DOT.GOV"  
<BeckhamRE@cdm.co <RANEY@VOLPE.DOT.GOV>, "mgoldade@peakpeak.com"  
<mgoldade@peakpeak.com>  
m> cc: "Autio, Anni" <AutioAH@cdm.com>  
Subject: FW: LB-000015, LB-000016, and LB-000017  
04/01/03 08:47 AM

For your review and approval.

- Richard Beckham

-----Original Message-----

**From:** Mahoney, Ron [mailto:Rmahoney@EMSL.com]  
**Sent:** Monday, March 31, 2003 6:11 PM  
**To:** Beckham, Richard

Subject: LB-000015, LB-000016, and LB-000017

Richard,

These should be final. The only recent revision is the addition of the Effective Date. These need to go to Mark and Mary for their final blessing.

<<LB-000015(rev 3\_31\_03).doc>> <<LB-000016 rev. (3\_31\_03).doc>>  
<<LB-000017 rev(3\_31\_03).doc>>

R.K. Mahoney  
Senior Analyst  
Special Projects Coordinator  
EMSL Analytical, Inc.  
Westmont, NJ  
800.220.3675, x1218  
mahoney@emsl.com

(See attached file: LB-000015(rev 3\_31\_03).doc)(See attached file:  
LB-000016 rev. (3\_31\_03).doc)(See attached file: LB-000017  
rev(3\_31\_03).doc)



## Request for Modification

To  
Laboratory Activities  
LB-000019

**Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.  
File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:**

All Labs Applicable forms – copies to: EPA, Volpe, CDM, All project labs

Individual Labs Applicable forms – copies to: EPA, Volpe, CDM, Initiating Lab

Method (circle one/those applicable): TEM-AHERA, TEM-ISO 10312, PCM-NIOSH 7400, PLM-NIOSH 9002,  
EPA/600/R-93/116, ASTM D5755-95, EPA/540/2-90/005a, Other: All TEM Methodologies

Requester: R. K. Mahoney

Title: Senior Analyst/Special Projects Coordinator

Company: EMSL Analytical, Inc.

Date: 21 January 2003

### Description of Modification:

Clarification of bench sheet recording format for grid openings in which no countable structures are recorded.

### Reason for Modification:

The electronically deliverable spread sheet for TEM analysis developed for the Libby project requires "ND" (None Detected) to be entered for grid openings in which no countable structures are recorded. The ND code has been used on all electronic deliverables for the Libby project. The code "NSD" (No Structure Detected) has been used on hand written bench sheets up until this date. As of 21 January 2003, "ND" will be used on the bench sheets as well as the electronically deliverables.

### Potential Implications of this Modification:

There are no potential negative implications resulting from this clarification of terms.

Laboratory Applicability (circle one): All Individual(s) EMSL Analytical, Inc.

### Duration of Modification (circle one):

Temporary Date(s): \_\_\_\_\_

Analytical Batch ID: \_\_\_\_\_

Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

Permanent

(Complete Proposed Modification Section)

Effective Date: 21 January 2003

Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable): \_\_\_\_\_

Technical Review: R. K. Mahoney  
(Laboratory Manager or designate)

Date: 27 March 2003

Project Review and Approval: Mark Raney  
(Volpe: Mark Raney)

Date: 7 March 2003

Approved By: Mary Goldade

Date: 7 March 2003

Title: EPA Regional Chemist  
(USEPS: Mary Goldade)

**Mahoney, Ron**

---

**From:** Raney, Mark [RANEY@VOLPE.DOT.GOV]  
**Sent:** Friday, March 07, 2003 2:50 PM  
**To:** 'Beckham, Richard'; 'Charlie LaCerra'; 'rdemalo@emsl.com'; 'rmahoney@emsl.com'; Autio, Anni; Raney, Mark; 'bratlin@syrrs.com'; 'Goldade.mary@EPAMail.epa.gov'; Montera, Jeff  
**Subject:** RE: MOD LB-000019

I find Laboratory Request for Modification # LB-000019 acceptable as written and here by provide Volpe approval to this MOD.

Richard, Please make sure MOD IDs get inserted onto the mod forms themselves (not just the file ID), so you will be able to identify the IDs based upon hardcopy alone. Also, even though this MOD is applicable to an individual lab, all MODs are to be forwarded to all labs for informational purposes and to give them an opportunity to provide comments. All labs however are REQUIRED to provide comments to only MODs that are applicable to all labs.

Mark Raney  
Environmental Engineer

US DOT / Volpe Center  
Environmental Engineering Division, DTS-33  
phone: 617-494-2377  
cell: 617-694-8223  
fax: 617-494-2789  
raney@volpe.dot.gov

-----Original Message-----

**From:** Beckham, Richard [mailto:BeckhamRE@cdm.com]  
**Sent:** Thursday, March 06, 2003 9:54 AM  
**To:** 'Charlie LaCerra'; 'rdemalo@emsl.com'; 'rmahoney@emsl.com'; Autio, Anni; 'Raney@volpe.dot.gov'; 'bratlin@syrrs.com'; 'Goldade.mary@EPAMail.epa.gov'; Montera, Jeff  
**Subject:** MOD LB-000019

This MOD impacts only EMSL. For your review and comment:

<<LB-000019.doc>>  
- Richard Beckham

**Mahoney, Ron**

---

**From:** Mary Goldade [mgoldade@peakpeak.com]  
**Sent:** Friday, March 07, 2003 12:29 PM  
**To:** Raney, Mark  
**Cc:** Jeff G. Montera; rmahoney@emsl.com; Autio, Anni; William Brattin; Goldade.Mary@epamail.epa.gov  
**Subject:** Re: MOD LB-000019

I agree that this mod form is acceptable, and should be discussed on the next lab call to be certain similar issues are not encountered at other labs.

Mary

----- Original Message -----

**From:** "Raney, Mark" <RANEY@VOLPE.DOT.GOV>  
**To:** "Goldade, Mary (HOME)" <mgoldade@peakpeak.com>  
**Sent:** Friday, March 07, 2003 10:18 AM  
**Subject:** FW: MOD LB-000019

>  
> FYI  
>  
>  
> -----Original Message-----  
> **From:** Beckham, Richard [mailto:BeckhamRE@cdm.com]  
> **Sent:** Thursday, March 06, 2003 9:54 AM  
> **To:** 'Charlie LaCerra'; 'rdemalo@emsl.com'; 'rmahoney@emsl.com'; Autio,  
> Anni; 'Raney@volpe.dot.gov'; 'brattin@syres.com';  
> 'Goldade.mary@EPAMail.epa.gov'; Montera, Jeff  
> **Subject:** MOD LB-000019  
>  
>  
> This MOD impacts only EMSL. For your review and comment  
>  
> <<LB-000019.doc>>  
> - Richard Beckham  
>  
>  
>

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## Request for Modification

To

Laboratory Activities

LB-000028

**Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.**

**File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:**

All Labs Applicable forms – copies to: EPA, Volpe, CDM, All project labs

Individual Labs Applicable forms – copies to: EPA, Volpe, CDM, Initiating Lab

Method (circle one/those applicable): TEM-AHERA, TEM-ISO 10312, PCM-NIOSH 7400, PLM-NIOSH 9002, EPA/600/R-93/116, ASTM D5755-95, EPA/540/2-90/005a, Other: All TEM Methodologies

Requester: R. K. Mahoney Title: Senior Analyst / Special Projects Coordinator  
Company: EMSL Analytical, Inc. Date: 17 June 2003

### Description of Modification:

This is a clarification pertaining to the re-analysis of TEM samples when some of the originally read grid openings in a sample selected for re-analysis have become unreadable. In the event that more than half of the originally read grid openings have become unreadable, select the closest adjacent sample from the same sample delivery group with adequate intact grid openings for re-analysis. If half or less of the original openings on the sample selected are unreadable, make note in the Comments box in Data Entry 1 of the TEM EDD as to which grid openings are unreadable, and proceed with analysis of the original sample.

### Reason for Modification:

This clarification is intended to provide more complete TEM re-analysis data.

### Potential Implications of this Modification:

There are no negative implications to this clarification.

Laboratory Applicability (circle one): All Individual(s) \_\_\_\_\_

### Duration of Modification (circle one):

Temporary Date(s): \_\_\_\_\_  
Analytical Batch ID: \_\_\_\_\_

Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

Permanent (Complete Proposed Modification Section) Effective Date: 17 June 2003

Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):

Technical Review: R. K. Mahoney EMSL Date: 18 July 2003  
(Laboratory Manager or designate)

Project Review and Approval: Mark Ramey Date: 7/18/03  
(Volpe: Project Technical Lead or designate)

Approved By: Waleed Goldade Date: 6/24/03

Title: Project Chemist  
(USEPA/Project Chemist or designate)



Mary Goldade

06/24/03 01:20 PM

To: "Beckham, Richard" <BeckhamRE@cdm.com>  
cc: "Autio, Anni" <AutioAH@cdm.com>, "Bill Egeland" <begeland@mastest.com>, "Bob.Shumate@battaenv.com" <Bob.Shumate@battaenv.com>, "brattin@syrres.com" <brattin@syrres.com>, "Charlie LaCerra" <clacerra@emsl.com>, "corbin77@atc-enviro.com" <corbin77@atc-enviro.com>, "dmazzaferro@mastest.com" <dmazzaferro@mastest.com>, "Gustavo Delgado" <gdelgado77@atc-enviro.com>, "Garth B. Freeman" <gfreeman@mastest.com>, "jeanneorr@resienv.com" <jeanneorr@resienv.com>, "mgoldade@peakpeak.com" <mgoldade@peakpeak.com>, "m\_szynskie@resienv.com" <m\_szynskie@resienv.com>, "Naresh C. Batta" <ncbatta@battaenv.com>, "Raney@volpe.dot.gov" <Raney@volpe.dot.gov>, "rdemalo@emsl.com" <rdemalo@emsl.com>, "rhatfield@mastest.com" <rhatfield@mastest.com>, "rmahoney@emsl.com" <rmahoney@emsl.com>, "Shu-Chun Su" <scsu@delanet.com>, "William Longo" <wlongo@mastest.com>

Subject: Re: EPA Approved w/ revisions MOD LB-000028

EPA approves Mod LB-000028 with revisions as attached.



LB-000028 (MG 6-24-03).

**Mary Goldade**

**Regional Superfund Chemist**



U.S. Environmental Protection Agency, Region 8

999 19<sup>th</sup> Street, Suite 300

Mail Code: BEPR-PS

Denver, CO 80202

Phone: (303) 312-7024

Fax: (303) 312-6065

email: goldade.mary@epa.gov

"Beckham, Richard" <BeckhamRE@cdm.com>



"Beckham, Richard"  
<BeckhamRE@cdm.co  
m>

06/23/03 08:42 AM

To: "Charlie LaCerra" <clacerra@emsl.com>, "Charlie LaCerra" <clacerra@emsl.com>, "jeanneorr@resienv.com" <jeanneorr@resienv.com>, "rdemalo@emsl.com" <rdemalo@emsl.com>, "rmahoney@emsl.com" <rmahoney@emsl.com>, "William Longo" <wlongo@mastest.com>, "rhatfield@mastest.com" <rhatfield@mastest.com>, "Bill Egeland" <begeland@mastest.com>, "Bob.Shumate@battaenv.com" <Bob.Shumate@battaenv.com>, "Naresh C. Batta" <ncbatta@battaenv.com>, "Shu-Chun Su" <scsu@delanet.com>, "corbin77@atc-enviro.com" <corbin77@atc-enviro.com>, "Gustavo Delgado" <gdelgado77@atc-enviro.com>, "Garth B. Freeman" <gfreeman@mastest.com>, "Autio, Anni" <AutioAH@cdm.com>, "Raney@volpe.dot.gov" <Raney@volpe.dot.gov>, "brattin@syrres.com" <brattin@syrres.com>, Mary Goldade/EPR/R8/USEPA/US@EPA, "dmazzaferro@mastest.com" <dmazzaferro@mastest.com>, "mgoldade@peakpeak.com" <mgoldade@peakpeak.com>, "m\_szynskie@resienv.com" <m\_szynskie@resienv.com>

cc:

Subject: MOD LB-000028

This MOD impacts all labs. For your review and comment.

- Richard Beckham

<<LB-000028.doc>>

---

**From:** "LaCerra, Charles" <CLaCerra@EMSL.com>  
**To:** "Carr, Kim" <KCarr@EMSL.com>; "EMSL Mobile Lab - Asbestos" <mobileasbestoslab@EMSL.com>  
**Sent:** Friday, July 18, 2003 5:57 AM  
**Attach:** LB-000025\_rev (MG 6-04-03 email).doc; LB-000027 (MG 6-24-03).doc; LB-000028 (MG 6-24-  
**Subject:** FW: MODs: LB-000025, 26, 27 & 28

-----Original Message-----

**From:** Raney, Mark [mailto:RANEY@VOLPE.DOT.GOV]  
**Sent:** Friday, July 18, 2003 7:53 AM  
**To:** 'Beckham, Richard'; Autio, Anni  
**Cc:** 'Goldade, Mary'; 'Goldade, Mary (HOME)'; 'Orr, Jeaane at Reservoir  
Env'; 'Mahoney, Ron'; 'Demalo, Rob (EMSL)'; 'LaCerra, Charles'  
**Subject:** MODs: LB-000025, 26, 27 & 28

Richard,

LB-000025 (EMSL): Volpe provided approval (with revisions) on 6/18/03 & EPA approved on 5/14/03 (see emails and attachment below). I have yet to see a final version for signature. EMSL should finalize, sign and distribute for signature.

LB-000026 (EMSL): Approved and signed by both Volpe and EPA.

LB-000027 (RESI): MOD provided on 6/23/03 via Richard Beckham, Approved by EPA (with revisions) on 6/24/03. Volpe concurs with EPA and herby provides approval with EPA's revisions (see attached). RESI should finalize, sign and distribute for signature.

LB-000028 (EMSL): MOD provided on 6/23/03 via Richard Beckham, Approved by EPA (with revisions) on 6/24/03. Volpe concurs with EPA and herby provides approval with EPA's revisions (see attached). EMSL should finalize, sign and distribute for signature.

Please let me know if anyone has any questions.

Mark.

7/18/2003

—Original Message—

From: Beckham, Richard [mailto:BeckhamRE@cdm.com]  
Sent: Wednesday, July 16, 2003 5:30 PM  
To: 'RANEY@VOLPE.DOT.GOV'; Autio, Anni  
Subject: MOD Status

For MODs 27 and 28, I have email approvals from EPA, but have not been able to locate approvals from Volpe. CDM received a hardcopy of 27 with an original signature from RESI, that was subsequently forwarded to Volpe on 7/8/3. (Did I miss an approval email?) To my knowledge, a hardcopy of 28 has not been prepared.

- Richard Beckham

—Original Message—

From: Raney, Mark  
Sent: Wednesday, June 18, 2003 10:56 AM  
To: 'Mahoney, Ron'  
Cc: 'Anni Autio'; 'Mary Goldade'  
Subject: RE: EPA Markups: MOD LB-000025

Ron,

I concur with Mary's comments below. I provide Volpe's approval for MOD LB-000025 with Mary's changes and the addition of an estimate of the number of samples involved (i.e., < 20).

Thanks,

Mark.

—Original Message—

From: Mahoney, Ron [mailto:Rmahoney@EMSL.com]  
Sent: Wednesday, June 04, 2003 9:27 AM  
To: 'Mark Raney'

7/18/2003

Cc: 'Anni Autio'; 'Mary Goldade'; CDM STAFF  
Subject: FW: EPA Markups: MOD LB-000025

Mark,

Do you have any other comments for this mod? Mary asked for an estimate of the number of samples involved, and we agreed on < 20. The number is more likely < 10, but we've decided to err on the conservative side.

If I can get your input, we can put this one to bed.

R.K. Mahoney  
Senior Analyst  
Special Projects Coordinator  
EMSL Analytical, Inc.  
Westmont, NJ  
800.220.3675, x1218  
[rmahoney@emsl.com](mailto:rmahoney@emsl.com)

-----Original Message-----

From: Mary Goldade [<mailto:mgoldade@peakpeak.com>]  
Sent: Wednesday, May 14, 2003 6:32 PM  
To: Beckham, Richard; 'Charlie LaCerra'; [jeanneorr@resienv.com](mailto:jeanneorr@resienv.com); [rdemalo@emsl.com](mailto:rdemalo@emsl.com); [rmahoney@emsl.com](mailto:rmahoney@emsl.com); 'William Longo'; [rhatfield@mastest.com](mailto:rhatfield@mastest.com); 'Bill Egeland'; [Bob.Shumate@battaenv.com](mailto:Bob.Shumate@battaenv.com); 'Naresh C. Batta'; 'Shu-Chun Su'; [corbin77@atc-enviro.com](mailto:corbin77@atc-enviro.com); 'Gustavo Delgado'; 'Garth B. Freeman'; Autio, Anni; [Raney@volpe.dot.gov](mailto:Raney@volpe.dot.gov); [brattin@syrres.com](mailto:brattin@syrres.com); [Goldade.mary@EPAMail.epa.gov](mailto:Goldade.mary@EPAMail.epa.gov); [dmazzaferro@mastest.com](mailto:dmazzaferro@mastest.com); [m\\_szynskie@resienv.com](mailto:m_szynskie@resienv.com)  
Subject: EPA Markups: MOD LB-000025

Suggested changes to the MOD are attached.  
Ron-Do you already have in hand an estimate regarding the actual number of samples this affects (i.e., are you able to quantify the term "few/limited"?)  
Thanks,  
Mary

----- Original Message -----

From: "Beckham, Richard" <[BeckhamRE@cdm.com](mailto:BeckhamRE@cdm.com)>  
To: "Charlie LaCerra" <[clacerra@emsl.com](mailto:clacerra@emsl.com)>; <[jeanneorr@resienv.com](mailto:jeanneorr@resienv.com)>; <[rdemalo@emsl.com](mailto:rdemalo@emsl.com)>; <[rmahoney@emsl.com](mailto:rmahoney@emsl.com)>; "William Longo" <[wlongo@mastest.com](mailto:wlongo@mastest.com)>; <[rhatfield@mastest.com](mailto:rhatfield@mastest.com)>; "Bill Egeland" <[begeband@mastest.com](mailto:begeband@mastest.com)>; <[Bob.Shumate@battaenv.com](mailto:Bob.Shumate@battaenv.com)>; "Naresh C. Batta" <[ncbatta@battaenv.com](mailto:ncbatta@battaenv.com)>; "Shu-Chun Su" <[scsu@delanet.com](mailto:scsu@delanet.com)>;

7/18/2003

<corbin77@atc-enviro.com>; "Gustavo Delgado"  
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Sent: Wednesday, May 14, 2003 3:28 PM  
Subject: MOD LB-000025

> This MOD impacts only EMSL. For your review and comment:  
>  
> <<LB-000025.doc>>  
> - Richard Beckham

<<LB-000025\_rev (MG 6-04-03 email).doc>> <<LB-000027 (MG  
6-24-03).doc>> <<LB-000028 (MG 6-24-03).doc>>  
>  
>  
>

7/18/2003

**Mary Goldade**

**07/29/03 01:57 PM**

**To: Anni Autio**

**cc: Mark Raney**

**cc:**

**Subject: LB-000027 & LB-000028 are signed and mailed**

**Anni & Joe,**

**I have mail you the original copiew of the mods LB-000027 & LB-000028.**

**Several of the email approval pages were not provided. I attached them.**

---

**Mary Goldade**

**Regional Superfund Chemist**

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**Request for Modification**  
to  
**Laboratory Activities**  
LB-000029b

**Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.**  
**File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:**

All Labs Applicable forms – copies to: EPA, Volpe, CDM, All project labs  
Individual Labs Applicable forms – copies to: EPA, Volpe, CDM, Initiating Lab

Method (circle one/those applicable): TEM-AHERA TEM-ISO 10312 PCM-NIOSH 7400 NIOSH 9002  
EPA/600/R-93/116 ASTM D5755 EPA/540/2-90/005a SRC-LIBBY-03  
Other: \_\_\_\_\_

Requester: Lynn Woodbury Title: Technical consultant  
Company: Syracuse Research Corporation Date: December 7, 2006

**Description of Modification:**

Permanent clarifications to laboratory-based Quality Control (QC) sample analysis. The purpose of the attached is to standardize the frequency of analysis and procedures for interpretation of the results for laboratory-based Quality Control (QC) samples for TEM analyses of air and dust. The general concepts presented in this modification may also be used for soil and water, but specific details regarding the frequency and interpretation of laboratory QC samples will need to be adjusted for these media.

**Reason for Modification:**

This modification is needed to standardize the frequency with which different types of QC samples are prepared in different laboratories in the program, and to ensure that all results are evaluated in accord with a standard set of criteria.

**Potential Implications of this Modification:**

There are no potential negative implications resulting from this standardization of QC procedures.

Laboratory Applicability (circle one): All Individual(s) \_\_\_\_\_

**Duration of Modification (circle one):**

Temporary Date(s): \_\_\_\_\_  
Analytical Batch ID: \_\_\_\_\_

*Temporary Modification Forms -- Attach legible copies of approved form w/ all associated raw data packages*

Permanent (Complete Proposed Modification Section) Effective Date: \_\_\_\_\_  
*Permanent Modification Forms -- Maintain legible copies of approved form in a binder that can be accessed by analysts.*

Data Quality Indicator (circle one) – Please reference definitions on reverse side for direction on selecting data quality indicators:

Not Applicable Reject Low Bias Estimate High Bias No Bias

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable): \_\_\_\_\_

Technical Review: \_\_\_\_\_ Date: \_\_\_\_\_  
(Laboratory Manager or designate)

Project Review and Approval: \_\_\_\_\_ Date: 4/25/07  
(Volpe/Project Technical Lead or designate)

Approved By: W. J. Goldacke Date: 4/25/07  
(USEPA Project Chemist or designate)



## DATA QUALITY INDICATOR DEFINITIONS

**Reject** - Samples associated with this modification form are not useable. The conditions outlined in the modification form adversely effect the associated sample to such a degree that the data are not reliable.

**Low Bias** - Samples associated with this modification form are useable, but results are likely to be biased low. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated low.

**Estimate** - Samples associated with this modification form are useable, but results should be considered approximations. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimates.

**High Bias** - Samples associated with this modification form are useable, but results are likely to be biased high. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated high.

**No Bias** - Samples associated with this modification form are useable as reported. The conditions outlined in the modification form suggest that associated sample data are reliable as reported.

## ***QC Sample Type Definitions***

There are three categories of TEM laboratory QC samples: Blanks, Recounts, and Repreparations.

### **Blanks**

Lab Blank (LB) – This is a TEM grid that is prepared from a new, unused filter by the laboratory and is analyzed using the same procedure as used for field samples.

### **Recounts**

Recount Same (RS) – This is a TEM grid that is re-examined within the same laboratory and by the same microscopist who performed the initial examination. The microscopist examines the same grid openings as were counted in the original examination. Recount Same TEM analyses will be selected in accord with the procedure presented in Attachment 1.

Recount Different (RD) – This is a TEM grid that is re-examined within the same laboratory but by a different microscopist than who performed the initial examination. The microscopist examines the same grid openings as were counted in the original examination. Recount Different TEM analyses will be selected in accord with the procedure presented in Attachment 1.

Interlab (IL) - This is a TEM grid that is re-examined by a microscopist from a different laboratory than who performed the initial examination. The microscopist examines the same grid openings as were counted in the original examination. Interlab TEM analyses for air and dust will be selected in accord with the procedure presented in Attachment 2.

Verified Analysis (VA) – This is a recount of a TEM grid (same grid openings) performed in accord with the protocol for verified analysis as provided in NIST (1994) (provided as Attachment 3). Verified TEM analyses will be selected in accord with the procedure presented in Attachment 1.

### **Repreparations**

Repreparation (RP) – This is a TEM grid that is prepared from a new portion of the same filter that was used to prepare the original grid. Typically this is done within the same laboratory as did the original analysis, but a different laboratory may also prepare grids from a new piece of filter. Repreparations will be selected in accord with the procedure presented in Attachment 1.

## ***Frequency***

The minimum frequency for laboratory-based QC samples for TEM analyses (all media combined) shall be as follows:

QC Sample Type	Min. Frequency
Lab blank	4%
Recount same	1%
Recount different	2.5%
Verified analysis	1%
Repreparation	1%
Interlab	0.5%
<b>Total</b>	<b>10%</b>

Each laboratory should prepare and analyze lab blank, recount (same, different and verified), and reparation samples at the minimum frequency specified in the table above. The selection procedure and laboratory SOP for the selection of samples for the purposes of recounts and reparation are provided in Attachment 1. Samples for interlab comparisons will be selected by EPA's technical consultant (SRC) in accord with the selection procedure and laboratory SOP provided in Attachment 2.

### ***Procedure for Evaluating QC Samples and Responses to Exceptions***

The procedure for evaluating QC sample results varies depending on sample type. These procedures are presented below.

Note: The procedures for evaluating QC samples presented below are based in part on professional judgement and experience at the site to date. These procedures and rules for interpretation may be revised as more data are collected.

#### **Lab Blanks.**

There shall be no asbestos structure of any type detected in an analysis of 10 grid openings on any lab blank. If one or more asbestos structures are detected, the laboratory shall immediately investigate the source of the contamination and take immediate steps to eliminate the source of contamination before analysis of any investigative samples may begin.

#### **Recounts.**

All recount samples (same, different, verified, and interlab) will be evaluated by comparing the raw data sheets prepared by each analyst. Note that the raw data for samples must include sketches for both the initial and QC reanalysis, as described in modification LB-000030. All structure enumeration and measurements will adhere to the established project-specific documentation presented in LB-000016A and LB-000031A. The following criteria will be used to identify cases where results for LA structures are concordant (in agreement) or discordant (not in agreement). These LA criteria were established by microscopists experienced in the analysis of Libby amphibole asbestos, and serve as an initial attempt at review criteria developed using their professional experience. As the database continues to grow and we learn more, these criteria may be revisited and revised. Changes to the criteria for LA structures will be accompanied by scientific justification to support the change. Criteria for concordance on non-LA fibers (OA and C) fibers are the same as described in NIST (1994) (provided as Attachment 3).

Measurement parameter	Concordance Rule
Number of LA asbestos structures within each grid opening	For grid openings with 10 or fewer structures, counts must match exactly. For grid openings with more than 10 structures, counts must be within 10%.
Asbestos class of structure (LA, OA, C)	Must agree 100% on chrysotile vs. amphibole. For assignment of amphiboles to LA or OA bins, must agree on at least 90% of all amphibole structures.
LA Structure length	For fibers and bundles, must agree within 0.5 um or 10% (whichever is less stringent)  For clusters and matrices, must agree within 1 um or 20% (whichever is less stringent)
LA Structure width	For fibers and bundles, must agree within 0.5 um or 20% (whichever is less stringent).  For clusters and matrices, there is no quantitative rule for concordance.

Whenever a recount occurs in which there is one or more discordance, the sample will undergo verified analysis as described by NIST (1994), and the senior laboratory analyst will use the results of the validated analysis to determine the basis of the discordance, and will then take appropriate corrective action (e.g., re-training in counting rules, quantification of size, identification of types, etc). Whichever analytical result is determined to be correct will be identified with the word "Confirmed" in the sample comment field of the electronic data reporting sheet. In the special case where the original and the reanalysis are both determined to have one or more areas of discordance, a third electronic data report will be prepared that contains the correct results. This will be identified as QA Type = "Reconciliation". The laboratory should maintain records of all cases of discordant results and of actions taken to address any problems, in accord with the usual procedures and requirements of NVLAP. In addition, each laboratory should notify the CDM Laboratory Manager of any significant exceptions and corrective actions through a job-specific (temporary) modification form. The CDM Laboratory Manager will ensure that appropriate Volpe and EPA representatives are notified accordingly.

#### Repreparations.

Repreparation samples will be evaluated by comparing the total counts for the original and the re-preparation samples. In order to be ranked as concordant, the results must not be statistically different from each other at the 90% confidence interval, tested using the statistical procedure documented in Attachment 4. Whenever an exception is identified, a senior analyst shall determine the basis of the discordant results, and if it is judged to be related to laboratory procedures (as opposed to unavoidable variability in the sample), the laboratory shall then take appropriate corrective action (e.g., re-training in sample and filter preparation, counting rules, quantification of size, identification of types, etc).

#### Program-Wide Goals

While each laboratory shall monitor the results of the QC samples analyzed within their laboratory and shall take actions as described above, the overall performance of the program shall be monitored by assembling summary statistics on QC samples, combining data within and across laboratories. The program-wide goals shall be interpreted as follows:

QC Sample Type	Metric	Program-Wide Criteria		
		Good	Acceptable	Poor
Lab Blanks	% with $\geq 1$ asbestos structures	0% - 0.1%	0.2% - 0.5%	>0.5%
Recounts	Concordance on LA count	>95%	85-95%	<85%
	Concordance on type (chrysotile vs. amphibole)	>99%	95%-99%	<95%
	Concordance on LA length	>90%	80%-90%	<80%
	Concordance on LA width	>90%	80%-90%	<80%
Repreps	Concordance on LA concentration/loading	>95%	90-95%	<90%

As the database continues to grow and we learn more, these project-wide goals may be revisited and revised. Changes to the project-wide goals will be accompanied by appropriate justification to support the change.

#### REFERENCES

NIST. 1994. Airborne Asbestos Method: Standard Test method for Verified Analysis of Asbestos by Transmission Electron Microscopy – Version 2.0. National Institute of Standards and Technology, Washington DC. NISTIR 5351. March 1994.

## ATTACHMENT 1

### Selection Procedure and Laboratory SOP for Recounts (RS, RD, VA) and Repreparations (RP)

#### Selection Procedure

As specified in the Frequency section above, the frequency of Recount Same (RS) should be 1%, the frequency of Recount Different (RD) should be 2.5%, the frequency of Verified Analyses (VA) should be 1%, and the frequency of Repreparations (RP) should be 1%, corresponding to a total within-laboratory QC frequency of 5.5% for these analysis types. This is approximately 1 QC sample per 20 field samples. Based on this frequency, it is possible to determine which laboratory job(s) will have one or more samples selected for recount analysis or reparation.

For those laboratory jobs in which a recount or reparation sample is to be selected, the analyst should record the total number of structures observed in each sample. The sample(s) selected for recount or reparation should be those within the laboratory job with the highest number of structures per grid opening (GO) area examined (calculated as the number of GOs evaluated \* the GO area). When selecting samples for reparation, if possible, preferentially select samples in which the total number of GOs is 40 or less. Because reparation concordance is evaluated based on concentration, in order to achieve adequate statistical power, reparations must prepare and evaluate the same number of GOs as the original analysis to achieve a similar sensitivity. Hence, the selection of samples with 40 GOs or less will reduce analytical costs associated with reparations. When selecting samples for recount, it is not necessary to impose a minimum or maximum number of GOs because concordance is evaluated on a GO and structure basis, rather than a concentration basis. If all samples within the laboratory job are non-detect, a non-detect sample may be selected. A non-detect sample should be preferentially selected, every 10<sup>th</sup> selection.

This selection procedure will ensure that the recount analyses and reparations yield a dataset best suited to assess concordance<sup>1</sup>.

#### Laboratory SOP for Recount Analyses

1. For recount samples, re-analyze the selected sample in accord with the appropriate procedures for each type of recount (RS, RD, or VA). If more than 10 GOs were evaluated in the original analysis, the original analyst or laboratory director will select the 10 GOs with the highest number of structures to re-analyze in the recount analysis. The original analyst or laboratory director should also prepare a list of 5 alternate GOs, based on the next 5 GOs with the highest number of structures per GO area examined, which may be analyzed in the event that a selected GO is damaged and cannot be re-evaluated.
2. Record the results using the most recent version of the TEM data recording spreadsheet. Identify the Laboratory QC Type as "Recount Same", "Recount Different", or "Verified Analysis", as appropriate. Be sure that the grid and GO names match exactly with the names evaluated in the original analysis (including dashes, underscores, and spaces). If a GO cannot be evaluated (e.g., GO is damaged), DO NOT arbitrarily select a different GO for evaluation. Utilize the list of 5 alternative GOs provided by the original analyst or laboratory director to select an alternate GO for evaluation. Identify the names of any GOs that could not be evaluated in the comment field along with a brief description of why they could not be analyzed (e.g., grid opening F7 torn, not analyzed).
3. If there is one or more discordant GOs between the original analysis and the recount analysis, the sample will undergo verified analysis as described by NIST (1994), and the senior laboratory analyst will determine the basis of the discordance, and will then take appropriate corrective action (e.g., re-training in counting rules, quantification of size, identification of types, etc).

<sup>1</sup> It should be noted that this selection procedure will tend to result in the preferential selection of samples with the highest air concentration/dust loading values. Thus, summary statistics based on laboratory QC samples may tend to be biased high.

4. Submit the recount TEM spreadsheet to the CDM Laboratory Manager using standard deliverable procedures.

#### Laboratory SOP for Repreparations

1. Prepare 3 TEM grids using the standard preparation methods for air and dust at the Libby site.
2. Select two grids and read the same number of total GOs as the original analysis, using the TEM counting rules specified by the CDM Laboratory Manager. For example, if 40 GOs were evaluated in the original analysis, read 20 GOs from the first grid and 20 GOs from the second grid during the repreparation. Place the remaining grid in storage.
3. Record the results using the most recent version of the TEM data recording spreadsheet. Identify the QC Type as "Repreparation".
4. Submit the TEM spreadsheet to the CDM Laboratory Manager using standard deliverable procedures.

## ATTACHMENT 2

### Selection Procedure and Laboratory SOP for Interlabs (IL)

#### Selection Procedure

1. On the 1st of each month, EPA's technical consultant (SRC) will compile a list of all samples for which air and dust TEM results (ISO+AHRA+ASTM) were uploaded into Libby V2 Database in the preceding month (e.g., on November 1<sup>st</sup>, specify a date range of Oct 1-31, 2005). The Libby V2 Database query will be based on the upload date rather than the analysis date to ensure that analyses with an upload in a different month as the analysis date were not excluded<sup>2</sup>.
2. Identify the target number of air and dust interlab samples needed to meet the QC requirements for interlabs specified in the Frequency section above (0.5%). This is accomplished by multiplying the desired interlab frequency (0.5%) by the total number of air and dust analyses performed in the preceding month. For example, 178 TEM air analyses in October 2005 \* 0.5% = 0.89 (which is rounded up to 1). At a minimum, at least one air and one dust sample will be selected for interlab analysis.
3. For each medium (air and dust), rank order the TEM analyses from the preceding month on the total number of LA structures per GO area examined (calculated as the number of GOs evaluated \* the GO area). Selecting from analyses with a high number of LA structures per GO area examined increases the likelihood that the GOs evaluated as part of the interlab analysis will have one or more LA structures.
4. Exclude samples in which the total number of GOs is more than 40 GOs<sup>3</sup>. Exclude any samples that have already been selected for interlab evaluation previously.
5. Select the appropriate number of air and dust interlab samples from the available TEM analyses for which the total number of LA structures per GO area examined is higher than 0 (i.e., LA.detects). If the total number of samples with LA detects is equal to the desired number of interlab samples, select all detected samples for interlab analysis. If the total number of samples with LA detects is less than the desired number of interlab samples, select non-detect samples for interlab analysis. If the total number of samples with LA detects is higher to the desired number of samples, interlab samples will be selected to represent multiple laboratories, selecting those samples with the highest number of LA structures per GO examined first. EPA's technical consultant (SRC) will keep a running total of the number of samples selected by laboratory to ensure that the long-term frequency of interlabs for each laboratory is generally similar.
6. Submit list of selected interlab samples to the CDM Laboratory Manager.
7. Each month, the CDM Laboratory Manager will provide each laboratory with the list of samples selected for Interlab analysis.

<sup>2</sup> Consider the case where the TEM analysis for sample X-12345 was performed on September 22 and the results were uploaded on October 3. The interlab selection query performed on October 1, if limited to all results analyzed from September 1-30, would not capture the results for X-12345 because they had not yet been uploaded. The interlab selection query performed on November 1, limited to all results analyzed from October 1-31, would also not capture the results for sample X-12345 because the analysis date is outside of the specified range.

<sup>3</sup> Because all interlabs will be reprepared, these interlab reparation samples will also be evaluated for concordance with the original sample. Because reparation concordance is evaluated based on concentration, in order to achieve adequate statistical power, reparations must prepare and evaluate the same number of GOs as the original analysis to achieve a similar sensitivity. Hence, the focusing on samples with 40 GOs or less will reduce analytical costs associated with reparations.

## Laboratory SOP

### **At the Originating Laboratory:**

1. Upon receipt of the interlab sample list from the CDM Laboratory Manager, locate the appropriate sample filter. If less than ¼ of the sample filter is available, contact the CDM Laboratory Manager to identify an interlab replacement sample.
2. Prepare 3 TEM grids using the standard preparation methods for air and dust at the Libby site.
3. Select two grids and read the same number of total GOs as the original analysis, using the TEM counting rules specified by the CDM Laboratory Manager. For example, if 40 GOs were evaluated in the original analysis, read 20 GOs from the first grid and 20 GOs from the second grid during the reparation. Place the remaining grid in storage.
4. Record the orientation of each grid using the instructions for grid orientation specified in NVLAP (see Attachment 5).
5. When performing the TEM analysis, identify the relative position of each structure within the grid opening using the template provided as Attachment 6. It is not necessary to sketch the actual structure (as this is already recorded on the hard copy benchsheet), but the analyst should record the structure number which corresponds to the hard copy benchsheet. The analyst should also record the relative position of any non-asbestos mineral (NAM) structures. Use a new template for each grid opening.
6. Record the results using the most recent version of the TEM data recording spreadsheet. Identify the QC Type as "Reparation".
7. Submit the TEM spreadsheet to the CDM Laboratory Manager using standard deliverable procedures.
8. Identify which laboratory will perform the interlab analysis in accord with the following table:

Originating Lab	Lab for Interlab Sample #1	Lab for Interlab Sample #2	Lab for Interlab Sample #3	Lab for Interlab Sample #4	Lab for Interlab Sample #5	Lab for Interlab Sample #6...
Hygeia	Batta	MAS	RESI	EMSL-L	EMSL-W	Repeat... (beginning with the Lab identified for Sample #1)
Batta	MAS	RESI	EMSL-L	EMSL-W	Hygeia	
MAS	RESI	EMSL-L	EMSL-W	Hygeia	Batta	
RESI	EMSL-L	EMSL-W	Hygeia	Batta	MAS	
EMSL-L	EMSL-W	Hygeia	Batta	MAS	RESI	
EMSL-W	Hygeia	Batta	MAS	RESI	EMSL-L	

EMSL-L = EMSL, Mobile Lab in Libby

EMSL-W = EMSL, Westmont

9. If more than 10 GOs were evaluated in the reparation analysis, the reparation analyst or laboratory director will select the 10 GOs with the highest number of structures to re-analyze in the interlab analysis. The reparation analyst or laboratory director should also prepare a list of 5 alternate GOs, based on the next 5 GOs with the highest number of structures, which may be analyzed in the event that the selected GO is damaged and cannot be re-evaluated.
10. Ship the grid(s) for the interlab sample to the appropriate laboratory using standard chain of custody procedures. For each interlab sample, include a list of which GOs should be evaluated for each grid. The names of the grid and GOs provided on the chain of custody form should match exactly with those recorded in the original TEM data recording spreadsheet (including dashes, underscores, and spaces).
11. After the interlab laboratory has completed the interlab analysis, it will request copies of the hard copy laboratory benchsheet(s), the grid opening sketches, and TEM file for each interlab sample.



12. If areas of discordance are noted, the senior laboratory analyst from the interlab laboratory will contact the originating laboratory to discuss the basis of the discordance. As needed, the senior laboratory analyst will then take appropriate corrective action (e.g., re-training in counting rules, quantification of size, identification of types, etc).

#### **At the Interlab Laboratory:**

1. For each grid provided for interlab analysis, place the grid into the TEM grid holder ensuring that the grid orientation matches that which was specified by the originating laboratory (see Attachment 5 for details).
2. For the 10 GOs identified for interlab analysis, perform TEM analysis using the analysis method and counting rules specified on the chain of custody. Be sure that the grid and GO names match exactly with the names provided on the chain of custody (including dashes, underscores, and spaces). If a GO cannot be evaluated (e.g., GO is damaged), DO NOT arbitrarily select a different GO for evaluation. Utilize the list of 5 alternative GOs provided by the originating laboratory to select an alternate GO for evaluation. Identify the names of any GOs that could not be evaluated in the comment field along with a brief description of why they could not be analyzed (e.g., grid opening F7 torn, not analyzed).
3. When performing the TEM interlab analysis, identify the relative position of each structure within the grid opening using the template provided as Attachment 6. It is not necessary to sketch the actual structure (as this is already recorded on the hard copy benchsheet), but the analyst should record the structure number which corresponds to the hard copy benchsheet. The analyst should also record the relative position of any non-asbestos mineral (NAM) structures. Use a new template for each grid opening.
4. Record the results using the most recent version of the TEM data recording spreadsheet. Identify the Laboratory QC Type as "Interlab".
5. Submit the TEM spreadsheet to the CDM Laboratory Manager using standard deliverable procedures.
6. Contact the originating laboratory to request copies of the hard copy laboratory benchsheet(s), grid opening sketches, and TEM file for each interlab sample.
7. Perform a verified analysis using the procedures presented in NIST (1994) (provided as Attachment 3).
8. Assess the between-laboratory concordance, both on a GO-by-GO basis and on a structure-by-structure basis, using the Libby-specific recount concordance rules. If areas of discordance are noted, the senior laboratory analyst will contact the originating laboratory to discuss the basis of the discordance. As needed, the senior laboratory analyst will then take appropriate corrective action (e.g., re-training in counting rules, quantification of size, identification of types, etc).
9. Summarize the results of the verified analysis and document any changes in laboratory procedures or analyst training that were implemented to address noted discordances. Provide a copy of this report to EPA Chemist and the CDM Laboratory Manager.
10. Ship the grid(s) back to the originating lab.

## **ATTACHMENT 3**

**Airborne Asbestos Method:  
Standard Test Method for Verified Analysis of Asbestos  
by Transmission Electron Microscopy-Version 2.0.  
NIST (1994)**

**NISTIR 5351**

# **Airborne Asbestos Method: Standard Test Method for Verified Analysis of Asbestos by Transmission Electron Microscopy - Version 2.0**

**Shirley Turner  
Eric B. Steel**

U.S. DEPARTMENT OF COMMERCE  
Technology Administration  
National Institute of Standards  
and Technology  
Microanalysis Research Group  
Surface and Microanalysis Science Division  
Chemical Science & Technology Laboratory  
Gaithersburg, MD 20899

March 1994



**U.S. DEPARTMENT OF COMMERCE  
Ronald H. Brown, Secretary**

**TECHNOLOGY ADMINISTRATION  
Mary L. Good, Under Secretary for Technology**

**NATIONAL INSTITUTE OF STANDARDS  
AND TECHNOLOGY  
Arati Prabhakar, Director**

## Preface

This Interagency Report (IR) is one of a series of IRs that will form the basis of a method for analysis of airborne asbestos by transmission electron microscopy. The form and style of the American Society for Testing and Materials (ASTM) was adopted as a standard format for this series of reports.

## 1. Scope

1.1 This test method describes a procedure for verified analysis of asbestos by transmission electron microscopy.

1.2 The method is applicable only when sufficient information has been collected during the analyses of a grid square so that individual asbestos structures can be uniquely identified.

1.3 The method is written for the analysis of a grid square by two TEM operators but can be used for more than two operators with slight modifications. Due to the analysis of a grid square by more than one TEM operator, the test method can be applied only when contamination and beam damage of particles are minimized. The two TEM operators can use the same TEM for the analysis or the analyses can be done on different TEMs (in the same or in different laboratories).

1.4 The method can be used with any set of counting rules applied by all analysts. Though the method describes verification of asbestos particles, the method can also be used for verification of analyses of nonasbestos particles if all analysts use the same counting rules.

## 2. Terminology

### 2.1 Definitions:

2.1.1 *TEM*--transmission electron microscope.

2.1.2 *grid square, grid opening*--an area on a grid used for analysis of asbestos by transmission electron microscopy.

2.1.3 *verified analysis*--a procedure in which a grid opening is independently analyzed for asbestos by two or more TEM operators and in which a comparison and evaluation of the correctness of the analyses are made by a verifying analyst. Detailed information -- including absolute or relative location, a sketch, orientation, size (length, width), morphology, analytical information and identification -- is recorded for each observed structure.

2.1.3.1 *Discussion*--Verified analysis can be used to determine the accuracy of operators and to determine the nature of problems that the analyst may have in performing accurate analyses. Verified counts can be used to train new analysts and to monitor the consistency of analysts over time.

### 2.2 Description of Terms Specific to This Standard:

2.2.1 *counting rules*--rules used to determine the amount of asbestos present in an asbestos-containing sample. Counting rules are a part of most methods for analysis of asbestos by transmission electron microscopy including the AHERA method and the ISO method (see definitions below).

2.2.2 *AHERA method*<sup>1</sup>--procedure for analysis of asbestos by transmission electron microscopy developed by the Environmental Protection Agency with subsequent modifications by the National Institute of Standards and Technology.

2.2.3 *ISO method*<sup>2</sup>--procedure for analysis of asbestos by transmission electron microscopy developed by the International Standards Organization.

2.2.4 *particle*--an isolated collection of material deposited on a grid or filter.

2.2.5 *structure*--a particle or portion of a particle that contains asbestos and that is considered countable under the method used for asbestos analysis. A structure is a basic unit used in many methods of asbestos analysis to report the amount of asbestos present in a particle.

2.2.6 *TEM operator, TEM analyst*--person that analyzes a grid square by transmission electron microscopy to determine the presence of asbestos.

2.2.7 *verifying analyst*--person that compares the analyses of a grid square by two or more TEM operators. The reported asbestos is compared on a structure-by-structure basis by the verifying analyst. Structures that are not matched are relocated and reanalyzed by the verifying analyst. The verifying analyst is

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<sup>1</sup>Code Fed. Reg. 1987, 52 (No. 210), 41826-41905.

<sup>2</sup>ISO 10312 1993, in press.

preferably not one of the TEM operators. If this cannot be avoided, the job of verifying analyst should be rotated between the TEM operators.

**2.2.8 TEM analysis form**--form on which the analysis of a grid square is recorded. The information recorded for a verified analysis should include at least a sketch of the structure and information related to the absolute or relative location, size, identification and analytical data for the reported structures.

**2.2.9 report form**--form on which the evaluation of verified analyses is summarized. The form should be identical to or include all information given in Figure X1.1 of Appendix X1.

**2.2.10 SR (structures reported)**--the number of structures reported by a TEM analyst.

**2.2.11 TP (true positive)**--structure that is: 1) reported by both TEM operators or 2) reported by one operator and confirmed by the verifying analyst, or 3) reported by neither TEM operator but is found by the verifying analyst. The three types of true positives are discussed in the next three terms.

**2.2.12 TPM (true positive-matched)**--structure that is reported on the TEM analysis forms of both TEM operators.

**2.2.12.1 Discussion**--To qualify as a match, the structures should be comparable in the following characteristics: 1) absolute or relative location, 2) appearance in the sketch, 3) orientation, 4) size (length, width), 5) morphology (shape, hollow tube), 6) analytical information (chemistry and/or diffraction data), and 7) identification. In addition, the structures should be reported as countable by both analysts.

**2.2.13 TPU (true positive-unmatched)**--structure that is reported on the TEM analysis form of only one operator and that is confirmed as countable by the verifying analyst.

**2.2.14 TPV (true positive found by verifying analyst)**--structure not found by the two TEM operators but found by the verifying analyst.

**2.2.15 TNS (total number of structures)**--the number of structures determined to be in a grid opening by verified analysis of the grid opening. This value corresponds to the number of unique true positives found by the TEM operators and the verifying analyst.

**2.2.15.1 Discussion**--The value for the total number of structures is not necessarily the actual number on the grid square because both the TEM analysts and the verifying analyst may have missed one or more structures. The probability of a missed structure, however, decreases with an increased number of analysts.

**2.2.16 FN (false negative)**--structure that has not been reported as countable by one of the TEM analysts. False negatives can be divided into two categories--type A and type B as discussed in the next two terms.

**2.2.17 FNA (false negative-type A)**--false negative that was recorded on a TEM analyst's TEM analysis form but not reported as a structure. Some reasons for this type of false negative include: 1) structure misidentified as nonasbestos, 2) confusion with the counting rules, 3) incorrect length determination.

**2.2.18 FNB (false negative-type B)**--false negative that was not recorded on a TEM analyst's TEM analysis form. A reason for this type of false negative is that a structure was missed by an analyst.

**2.2.19 FP (false positive)**--reported particle that is incorrectly identified as a structure. Some reasons for false positives include: 1) structures counted more than one time, 2) materials misidentified as asbestos, 3) confusion with the counting rules, 4) incorrect length determination.

**2.2.20 TN (true negative)**--reported particle that is correctly characterized as zero structures.

**2.2.21 NL (not located structure)**--structure reported on one TEM analyst's TEM analysis form that cannot be located by the verifying analyst.

**2.2.21.1 Discussion**--The value for NL should be zero for most verified analyses, especially if the grid has not been removed from the TEM between the two analysts' counts. If, however, a grid has been removed from an instrument, there is a small possibility of fiber loss.

**2.2.22 AMB (ambiguous structure)**--a structure that 1) is identified as a structure by only one TEM operator and 2) is found by the verifying analyst but cannot be unambiguously identified as a structure due to beam damage, contamination, or other factors.

### 3. Significance and Use

3.1 The analysis of asbestos by transmission electron microscopy is important for the determination of the cleanliness of air or water and for research purposes. Verified analyses provide more accurate values for the concentration of asbestos on a grid opening than obtained by other methods. The accuracy should increase with an increased number of analysts participating in the verified count.

3.2 The test method can be used as part of a quality assurance program for asbestos analyses and as a training procedure for new analysts. The values for TP/TNS and FP/TNS can be plotted vs time on control charts to show improvements or degradations in the quality of the analyses. Experienced analysts should attain TP/TNS values  $\geq 0.85$  and FP/TNS values  $\leq 0.05$ . The test method can be used to characterize the types and, in many cases, the causes of problems experienced by TEM analysts.

3.3 The average of values obtained for TP/TNS and FP/TNS can be used to determine the analytical uncertainty for routine asbestos analyses.

### 4. Procedure

NOTE 1-- This test method involves two TEM operators and a verifying analyst. The steps discussed in items 4.1 and 4.2 are to be followed by the person coordinating the analyses by the TEM operators. This person can be one of the TEM operators, the verifying analyst or an independent person (e.g., a quality assurance officer). The steps discussed starting with item 4.3 are to be followed by the verifying analyst.

4.1 Obtain analyses of a grid square for asbestos by two TEM operators. Conduct the analyses independently so that the second operator has no knowledge of the results obtained by the first operator.

4.1.1 Require that the TEM operators record on the TEM analysis form information related to the absolute location of the structures or conduct analyses so that the relative location of the structures can be compared.

NOTE 2-- The absolute location of the structures can be recorded by various means including use of a digital voltmeter or computer readable stepping motors to record the position of a structure. To preserve information about the relative location of the reported structures, the analyses must be conducted so that both analysts: 1) orient the grid in the TEM in the same fashion, 2) start the analysis from the same corner of the grid square, 3) initially scan in the same direction, and 4) scan the grid square in parallel traverses.

4.1.2 Require that the TEM operators record on the TEM analysis form a sketch of the structure, the dimensions of the structure, analytical data and whether the structure is countable. The sketch of the structure should include any nearby features that could aid in subsequent identification - for instance, nearby particles, sample preparation features or grid bars.

4.2 Submit the analyses of the two TEM operators to the verifying analyst.

NOTE 3-- The remainder of this section describes procedures to be followed by the verifying analyst. The procedure for comparison of the TEM analysis forms is given in items 4.3-4.6 and examples of comparisons of count sheets are given in Figs. X2.1-X2.9 of Appendix 2. Appendix 3 contains a summary of the comparison process (Fig. X3.1) and a flow chart for comparison of structures in the TEM (Fig. X3.2). The procedure for completion of the report form is given in item 4.7.

4.3 Compare the two TEM analysis forms on a structure-by-structure basis. If a match of asbestos structures is observed, label both sketches with a TPM(number) either in the sketch box or in a column specifically designated for verified counts. An example is given in Fig. X2.1 of Appendix X2.

NOTE 4-- The next step in the procedure (item 4.4) is optional. The most prudent approach is to examine unmatched structures in the TEM (item 4.5).

4.4 Determine if the status of any of the unmatched structures can be unambiguously decided by examining the TEM analysis forms. If there is ambiguity in determining the status of a structure, the verifying analyst must examine the structure in the TEM as described in items 4.5-4.6. The comparison of TEM analysis forms and labelling of unmatched structures can be relatively straight forward as shown in Fig. X2.2 - X2.4 of Appendix X2 or more complex as described in the next item.

4.4.1 For most cases, the identification of true positives, false positives and false negatives can be done on a structure-by-structure basis. This cannot be done, however, in cases where analysts determine different numbers of countable structures in an asbestos-containing particle. In such cases, both analysts should be assigned one TPM(number) for identifying the particle as containing countable asbestos. The remaining structures are assigned TPU, FP or FN depending on the particular situation. Examples of such cases are given in Fig. X2.5 and Fig. X2.6 of Appendix X2.

4.5 Determine the status of any remaining unlabelled structures by examining the grid square in the TEM. Examples of TEM analysis forms containing structures that must be examined by transmission electron microscopy are given in Figs. X2.7 - X2.9 of Appendix 2. For each unlabelled structure requiring examination by transmission electron microscopy, follow items 4.5.1-4.5.7 and 4.6 until the structure is labelled. If there is another unlabelled structure, go back to item 4.5.1 and repeat the procedure. Continue until all structures are labelled. A summary flow chart for examination by TEM is given in Fig. X3.2. The procedure and flowchart do not cover the counting discrepancy discussed in item 4.4.1. If such a situation is recognized, the verifying analyst should follow the procedure given in item 4.4.1 and in the examples in Figs. X2.5 and X2.6.

NOTE 5-- The procedure in items 4.5.1-4.5.7 should cover the great majority of cases encountered when attempting to determine the status of the structures. There may, however, be more complex situations not covered in the procedure. If so, the verifying analyst should apply the basic principles outlined in items 4.5.1-4.5.7 and 4.4.1.

4.5.1 Determine if the reported structure can be located. If the structure cannot be found, label the reported structure NL (place the label next to the sketch or in a column specifically designated for verified analyses).

4.5.2 If the reported structure is found, determine if a judgement can be made as to its countability. If the structure cannot be judged as to its countability due to beam damage, contamination or other factors, label the reported structure AMB.

4.5.3 If a judgement can be made as to the countability of the reported structure, determine if the structure is countable. If the reported structure is not countable, label it FP(number). A unique number is given to the FP label so that it can be specifically referred to in the report form. Optional: Check the other analyst's TEM analysis form. If the other analyst sketched the particle and correctly reported it as noncountable, label the particle TN(number). Note: The values for TN are not recorded on the report form.

4.5.4 If the reported structure is correctly identified as a structure, determine if it was reported as countable elsewhere on the same analyst's TEM analysis form (i.e., the analyst counted the structure twice). If it is a duplicate, label the reported structure FP(number).

4.5.5 If the reported structure is not a duplicate, label the structure TPU(number).

4.5.6 Determine if the other TEM operator recorded a sketch of the structure. If the other TEM operator did not report the structure on his/her TEM analysis form, place an FNB(number) on their TEM analysis form in the approximate location where the structure should have been found. The number should correspond to that given to the TPU on the first analyst's TEM analysis form.

4.5.7 If the other TEM operator recorded a sketch of the structure, label the sketch with an FNA(number). The number should correspond to that given to the TPU on the first analyst's TEM analysis form.

4.6 Countable asbestos structures reported by neither TEM operator but found by the verifying analyst in the course of examining a grid square should be recorded on a separate TEM analysis form and labelled



TPV(number). The TEM operators should be assigned an FNA(number) or FNB(number) as described in items 4.5.6-4.5.7.

4.7 Complete the report form as described in items 4.7.1-4.7.10.

4.7.1 Complete the heading of the report form and fill in the initials or names of the two TEM operators on the first line of the report form table.

4.7.2 Count the number of asbestos structures obtained by each analyst and enter the value as SR (structures reported) on the report form.

4.7.3 Determine the number of true positives that are matched (TPM), the number of true positives that are unmatched (TPU) and the total number of true positives (TP) obtained for each TEM operator on the grid square and enter the values on the report form.

4.7.4 Determine and record on the report form the number of true positives found by the verifying analyst (TPV).

4.7.5 Determine and record on the report form the total number of structures (TNS) on the grid square.

4.7.6 Determine and record on the report form for each operator the following: 1) the number of false positives (FP), 2) the number of false negatives (FN), 3) the number of false negatives of type A and type B (FNA, FNB), 4) the number of structures that were not located (NL) and 5) the number of ambiguous structures (AMB).

4.7.7 Determine and record the values for TP/TNS, FP/TNS to two decimal places.

4.7.8 List on the report form the suspected reasons for the false positives obtained by each analyst. Some examples would be as follows: incorrect length measurement, structures counted twice, problem with interpretation of the counting rules, misidentification of a structure.

4.7.9 List on the report form the suspected reasons for false negatives (FNA and FNB). Some examples would be: incorrect length measurement, problem with interpretation of the counting rules, misidentification of material as asbestos, possible loss of sense of direction, and insufficient overlap of traverses.

4.7.10 Append any other relevant comments to the report form (quality of the preparation, etc.).

4.8 Check the numbers on the report form using the equations given in the calculation section.

## 5. Calculation

5.1 The values on the report form should be consistent with the following equations:

For both analyses:

$$TNS = TPM + TPU(\text{Operator 1}) + TPU(\text{Operator 2}) + TPV$$

For a given analysis:

$$SR = TP + FP + NL + AMB$$

$$TP = TPM + TPU$$

$$FN = FNA + FNB$$

$$TNS = TP + FN$$

$$I = TP/TNS + FN/TNS$$

## 6. Precision and Bias

6.1 To determine the precision of the method, independent verified analyses were conducted by operators in two laboratories on a set of 21 grid squares. The mean value for TNS for the data set was 16.2 structures/grid square and the pooled standard deviation of the pairs of verified count determinations was 1.12 structures/grid square. The confidence at approximately the 95% level (2 standard deviations) of a reported verified count value in this data set is 2.24 structures/grid square or 13.9% of the mean value for TNS. We use 13.9% as an estimate of the imprecision of the method.

NOTE 6-- The differences in the values obtained for the independent verified analyses described in item 6.1 are, for the most part, due to differences in interpretation of the counting rules. The structures analyzed in the study were complex and therefore the imprecision estimate discussed above likely represents an upper bound to the imprecision for the method.

6.2 The bias in the method will vary depending upon interpretation of the counting rules used in the analysis by the TEM operators and verifying analyst.

## 7. Keywords

7.1 asbestos; quality assurance; transmission electron microscopy; verified analysis

## APPENDIXES

(Nonmandatory Information)

## X1. TEST REPORT FORM

Fig. X1.1 The following format is suggested for use by the verifying analyst to report the comparison of the TEM operators' TEM analysis forms.

Grid box: \_\_\_\_\_

Date: \_\_\_\_\_

Grid slot: \_\_\_\_\_

Verifying Analyst: \_\_\_\_\_

Grid square: \_\_\_\_\_

	Analysis 1	Analysis 2
TEM Operator		
Structures Reported (SR)		
True Positives (TP)		
*TPM		
TPU		
*TPV		
*Total # Structures (TNS)		
False Positives (FP)		
False Negatives (FN)		
FNA		
FNB		
Not Located (NL)		
Ambiguous (AMB)		
TP/TNS		
FP/TNS		

\*The values for these items will be the same for both analyses.

## Test Report Form (continued)


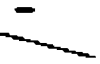

1) List details of suspected reasons for false positives. For each analyst describe reasons for FP1, FP2, FP3, etc. Note - it may not be possible to determine the reason for false positives for some structures.

2) List details of suspected reasons for false negatives (type A and type B). For each analyst describe reasons for FNA1, FNA2, etc.; FNB1, FNB2, etc. Note - it may not be possible to determine the reasons for false negatives for some structures.

## X2. EXAMPLES OF COMPARISONS OF TEM ANALYSIS FORMS

[Note: The TEM analysis forms shown in the examples are abbreviated and do not contain analysis information. The AHERA counting rules (1987) were used for all analyses.]

**Analyst 1**

Length ( $\mu\text{m}$ )	Width ( $\mu\text{m}$ )	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
0.7	0.1		TPM2	1	Chr
1.0	0.1		TPM3	1	Chr

**Analyst 2**








Length ( $\mu\text{m}$ )	Width ( $\mu\text{m}$ )	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
1.0	0.1		TPM3	1	Chr
0.7	0.1		TPM2	1	Chr

Fig. X2.1 Example of matching structures on two TEM analysis forms (refer to item 4.3 of the procedure). Three structures on a grid square were found by both analysts. The relative order of the last two structures is different on the two TEM analysis forms; this may be due to the nature of the traverses by the analysts. Matching structures are indicated by TPM(number).

Analyst 1

Length ( $\mu\text{m}$ )	Width ( $\mu\text{m}$ )	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
0.7	0.1		TPM2	1	Chr
1.0	0.1		TPM3	1	Chr
0.7	0.1		FP1	1	Chr

Analyst 2





Length ( $\mu\text{m}$ )	Width ( $\mu\text{m}$ )	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
1.0	0.1		TPM3	1	Chr
0.7	0.1		TPM2	1	Chr

Fig. X2.2 Example of determining the status of an unmatched structure from TEM analysis forms (refer to item 4.4 of the procedure). Three of the structures match in the two analyses. The last structure of analyst 1 is unmatched but can be seen from the TEM analysis form to be a duplicate of the second structure obtained by the same analyst (the two structures have the same identification, dimensions, orientation and a similar nearby particle). The duplicate structure is therefore assigned an FP1.

Analyst 1

Length ( $\mu\text{m}$ )	Width ( $\mu\text{m}$ )	Sketch	Verification	# Structures	ID
0.6	0.1		TPU1	1	Chr

Analyst 2



Length ( $\mu\text{m}$ )	Width ( $\mu\text{m}$ )	Sketch	Verification	# Structures	ID
0.6	0.1		FNA1	0	Chr

Fig. X2.3 Example of determining the status of unmatched structures from TEM analysis forms (refer to item 4.4 of the procedure). Both analysts have found the same particle as indicated by the dimensions, identification and orientation of the structure. However, analyst 2 has reported that the particle is not a structure (the cause of this oversight is not known). Analyst 1 is assigned a TPU1 and analyst 2 an FNA1.

Analyst 1

Length ( $\mu\text{m}$ )	Width ( $\mu\text{m}$ )	Sketch	Verification	# Structures	ID
0.4	0.1		FP1	1	Chr

Analyst 2



Length ( $\mu\text{m}$ )	Width ( $\mu\text{m}$ )	Sketch	Verification	# Structures	ID
0.4	0.1		TN1	0	Chr

Fig. X2.4 Example of determining the status of unmatched structures from TEM analysis forms (refer to item 4.4 of the procedure). Both analysts have found the same particle as indicated by the dimensions, identification and orientation of the particle on both TEM analysis forms. However, analyst 1 has reported that the particle is a structure (the cause of this oversight is not known). Analyst 1 is assigned an FP1 and analyst 2 a TN1.



Analyst 1

Length ( $\mu\text{m}$ )	Width ( $\mu\text{m}$ )	Sketch	Verification	# Structures	ID
1	0.6		TPM1 FNA1	1	Chr

Analyst 2

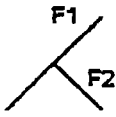

Length ( $\mu\text{m}$ )	Width ( $\mu\text{m}$ )	Sketch	Verification	# Structures	ID
					
1	0.1	F1	TPM1	1	Chr
0.6	0.1	F2	TPU1	1	Chr

Fig. X2.5 Example of determining the status of unmatched structures from TEM analysis forms (refer to item 4.4.1 of the procedure). Both analysts have found the same asbestos-containing particle as indicated by the dimensions, identification, and orientation of the particle. However, analyst 1 has reported one countable structure and analyst 2 has reported two countable structures. Under the AHERA counting rules, analyst 2 is correct. The structure reported by analyst 1 is assigned both a TPM1 and an FNA1. The two structures reported by analyst 2 are assigned a TPM1 and a TPU1, respectively.

## Analyst 1

Length ( $\mu\text{m}$ )	Width ( $\mu\text{m}$ )	Sketch	Verification	# Structures	ID
5	3		TPM1	1	Chr

## Analyst 2

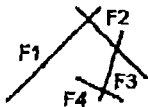


Length ( $\mu\text{m}$ )	Width ( $\mu\text{m}$ )	Sketch	Verification	# Structures	ID
					
5	0.1	F1	TPM1	1	Chr
3	0.1	F2	FP1	1	Chr
2	0.1	F3	FP2	1	Chr
1	0.1	F4	FP3	1	Chr

Fig. X2.6 Example of determining the status of unmatched structures from TEM analysis forms (refer to item 4.4.1 of the procedure). Both analysts have found the same asbestos-containing particle as indicated by the dimensions, identification, and orientation of the particle. However, analyst 1 has reported one structure and analyst 2 has reported four structures. Under the AHERA counting rules, analyst 1 is correct. The structure reported by analyst 1 is assigned a TPM1. The first structure reported by analyst 2 is labelled TPM1 and the remaining three reported structures are labelled FP1-FP3.


## Analyst 1


Length ( $\mu\text{m}$ )	Width ( $\mu\text{m}$ )	Sketch	Verification	# Structures	ID
0.4	0.1			0	Chr

## Analyst 2


Length ( $\mu\text{m}$ )	Width ( $\mu\text{m}$ )	Sketch	Verification	# Structures	ID
0.6	0.1			1	Chr


a

Length ( $\mu\text{m}$ )	Width ( $\mu\text{m}$ )	Sketch	Verification	# Structures	ID
0.4	0.1		FNA1	0	Chr

Length ( $\mu\text{m}$ )	Width ( $\mu\text{m}$ )	Sketch	Verification	# Structures	ID
0.6	0.1		TPU1	1	Chr

b




Length ( $\mu\text{m}$ )	Width ( $\mu\text{m}$ )	Sketch	Verification	# Structures	ID
0.4	0.1		TN1	0	Chr

Length ( $\mu\text{m}$ )	Width ( $\mu\text{m}$ )	Sketch	Verification	# Structures	ID
0.6	0.1		FP1	1	Chr



c

Fig. X2.7 Example of unmatched structures that must be examined by TEM (refer to item 4.5 of the procedure). a) Both analysts have likely found the same asbestos-containing particle as indicated by the identification and orientation of the fiber and by the presence of a similar particle nearby. However, the dimensions reported by the analysts differ and analyst 1 has reported zero structures and analyst 2 has reported one structure. The verifying analyst should determine the correct length of the fiber and determine if it qualifies as a structure. b) One possible outcome is that the verifying analyst finds that analyst 2 is correct. Analyst 2 is assigned a TPU1 and analyst 1 an FNA1. c) A second possible outcome is that the verifying analyst finds that analyst 2 is correct. Analyst 1 is assigned a TN1 and analyst 2 an FP1.

Analyst 1

Length (um)	Width (um)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
0.6	0.1			1	Chr
1.0	0.1		TPM2	1	Chr




Analyst 2

Length (um)	Width (um)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
1.0	0.1		TPM2	1	Chr



a

Fig. X2.8 Example of unmatched structures that must be examined by TEM (refer to item 4.5 of the procedure). a) Analyst 1 has reported one structure that analyst 2 has not reported. The verifying analyst should attempt to find the particle and determine if it qualifies as a structure. b) One possible outcome is that the verifying analyst finds that analyst 1 is correct. Analyst 1 is assigned a TPU1 and analyst 2 is assigned an FNB1. c) Another possible outcome is that the reported structure is not located. Analyst 1 is assigned an NL. Other possibilities (not illustrated) are that analyst 1 is incorrect (the particle is then labelled FP) or that the structure is too contaminated for characterization (the particle is then labelled AMB).




Analyst 1



Length (um)	Width (um)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
0.6	0.1		TPU1	1	Chr
1.0	0.1		TPM2	1	Chr

Analyst 2

Length (um)	Width (um)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
1.0	0.1		FNB1 TPM2	1	Chr

b


Length (um)	Width (um)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
0.6	0.1		NL1	1	Chr
1.0	0.1		TPM2	1	Chr

Length (um)	Width (um)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
1.0	0.1		TPM2	1	Chr

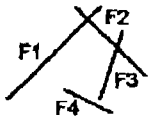
c

Fig. X2.8 (caption on previous page).

Analyst 1

Length ( $\mu\text{m}$ )	Width ( $\mu\text{m}$ )	Sketch	Verification	# Structures	ID
5	3			1	Chr


Analyst 2

Length ( $\mu\text{m}$ )	Width ( $\mu\text{m}$ )	Sketch	Verification	# Structures	ID
					
5	0.1	F1		1	Chr
3	0.1	F2		1	Chr
2	0.1	F3		1	Chr
1	0.1	F4		1	Chr

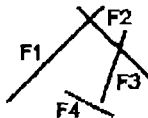
a

Fig. X2.9 Example of unmatched structures that must be examined by TEM (refer to item 4.5 of the procedure). a) Both analysts have likely found the same particle as indicated by the identification and orientation of the fibers. However, analyst 1 has recorded all fibers as touching (or intersecting) and has therefore counted the fiber arrangement as one structure under the AHERA method. Analyst 2 has reported four structures. The verifying analyst should find and examine the arrangement in the TEM to determine if the fiber labelled as F4 by analyst 2 is touching or intersecting the fiber labelled as F3. b) One possible outcome is that the verifying analyst finds that analyst 1 is correct. Analyst 1 is then assigned a TPM1 and analyst 2 is assigned a TPM1 and three FPs. Other possibilities (not illustrated) are that analyst 2 is correct (the structures reported by analyst 2 are then assigned a TPM and 3 TPUs and the structure reported by analyst 1 is assigned a TPM) or that the particle is too contaminated for identification (the structure reported by analyst 1 is then assigned a TPM and those reported by analyst 2 are assigned a TPM and three AMBs).

## Analyst 1

Length ( $\mu\text{m}$ )	Width ( $\mu\text{m}$ )	Sketch	Verification	# Structures	ID
5	3		TPM1	1	Chr

## Analyst 2

Length ( $\mu\text{m}$ )	Width ( $\mu\text{m}$ )	Sketch	Verification	# Structures	ID
					
5	0.1	F1	TPM1	1	Chr
3	0.1	F2	FP1	1	Chr
2	0.1	F3	FP2	1	Chr
1	0.1	F4	FP3	1	Chr

b

Fig. X2.9 (caption on previous page)

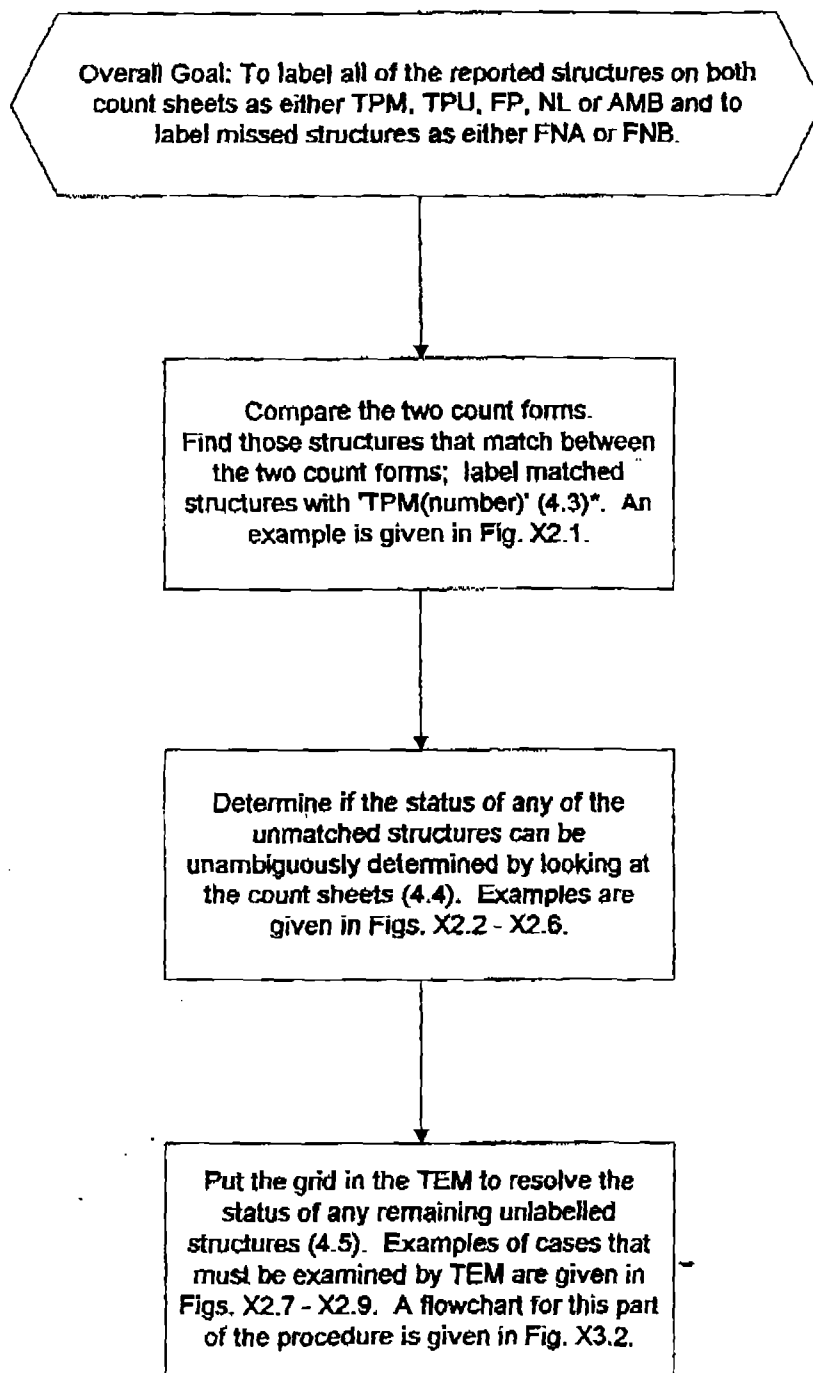
**X3. SUMMARY OF THE PROCEDURE FOR COMPARISON OF TWO TEM ANALYSIS FORMS**

Fig. X3.1 Summary of the overall procedure for comparison of TEM analysis forms by the verifying analyst.

\*Numbers in parentheses in each block refer to the item number in the procedure.



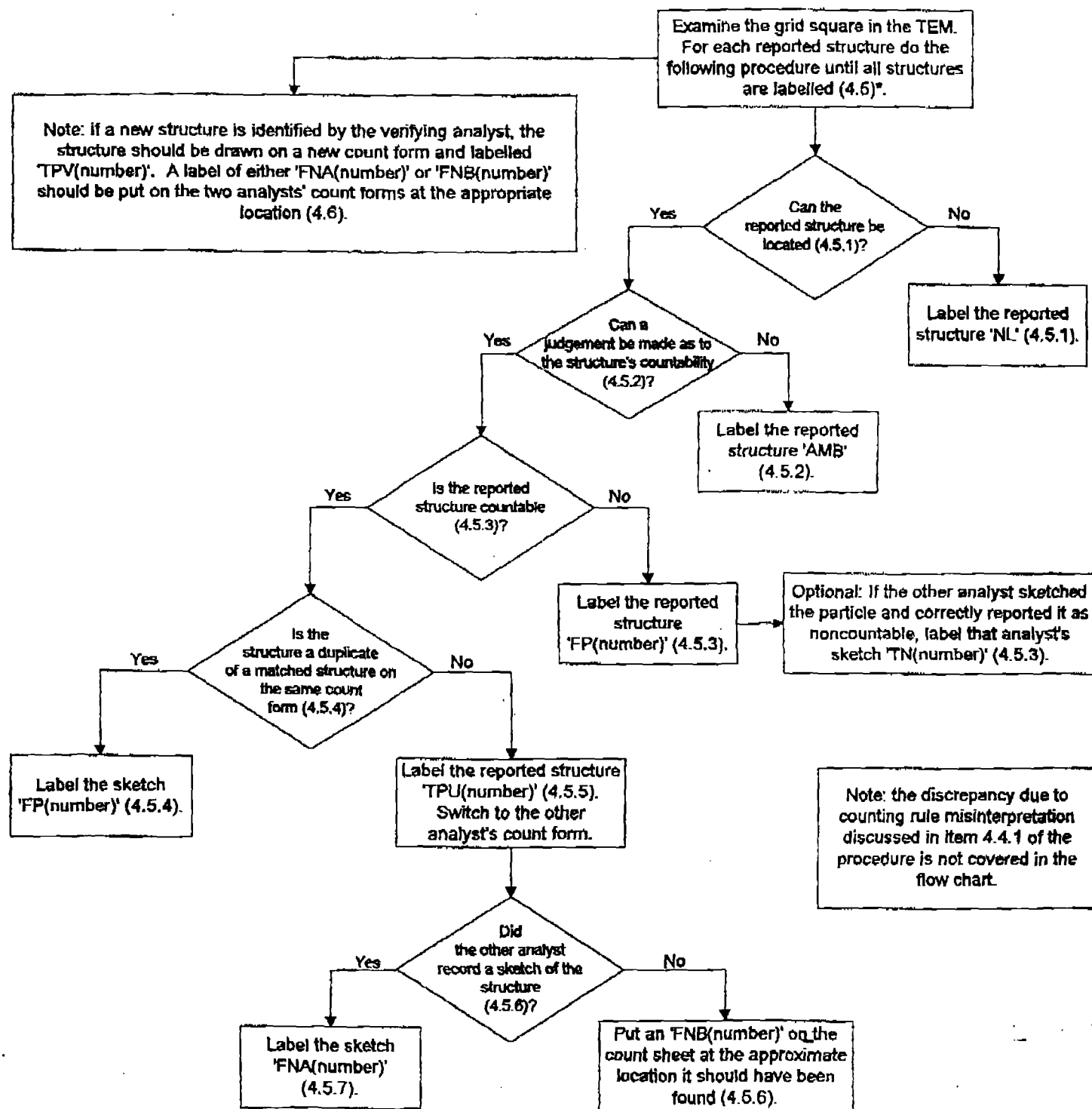


Fig. X3.2 Flowchart for examination of a structure in the TEM. The flowchart is an expansion of the last block in Fig. X3.1. \*Numbers in parentheses in each block refer to the item number in the procedure.

## ATTACHMENT 4

### Statistical Comparison of Two Poisson Rates

#### 1.0 INTRODUCTION

An important part of the Quality Control plan for this project is the reparation and reanalysis of a number of TEM grids for quantification of asbestos fiber concentrations in air and dust. Because of random variation, it is not expected that results from reparations samples should be identical. This attachment presents the statistical method for comparing two measurements and determining whether they are statistically different or not.

#### 2.0 STATISTICAL METHOD

This method is taken from "Applied Life Data Analysis" (Nelson 1982). Input values required for the test are as follows:

N1 = Fiber count in first evaluation  
S1 = Sensitivity of first evaluation  
N2 = Fiber count in second evaluation  
S2 = Sensitivity of second evaluation

The test is based on the confidence interval around the ratio of the two observed Poisson rates:

Rate 1 =  $N1 \cdot S1$   
Rate 2 =  $N2 \cdot S2$   
Ratio = Rate 1 / Rate 2

$$\text{Lower Bound} = \left( \frac{S1}{S2} \right) \left( \frac{N1}{N2 + 1} \right) / F \left[ \frac{1 + \gamma}{2}; 2 \cdot N2 + 2, 2 \cdot N1 \right]$$

$$\text{Upper Bound} = \left( \frac{S1}{S2} \right) \left( \frac{N1 + 1}{N2} \right) \cdot F \left[ \frac{1 + \gamma}{2}; 2 \cdot N1 + 2, 2 \cdot N2 \right]$$

where  $\gamma$  is the confidence interval (e.g., 0.95) and  $F[\delta; df1, df2]$  is the 100 $\delta$ th percentile of the F distribution with  $df1$  degrees of freedom in the numerator and  $df2$  degrees of freedom in the denominator.

If the lower bound of the ratio is  $> 1$ , then it concluded that rate 1 is greater than rate 2 at the 100(1- $\gamma$ )% significance level. If the upper bound of the ratio is  $< 1$ , then it concluded that rate 1 is less than rate 2 at the 100(1- $\gamma$ )% significance level. Otherwise, it is concluded that rate 1 and rate 2 are not different from each other at the 100(1- $\gamma$ )% significance level.

#### Example:

N1 = 4 structures  
S1 = 0.0001 (cc)<sup>-1</sup>  
Rate 1 = 4 · 0.0001 = 0.0004 s/cc

N2 = 6 structures  
S2 = 0.001 (cc)<sup>-1</sup>  
Rate 2 = 6 · 0.001 = 0.006 s/cc

$\gamma = 0.95$

$$\text{Lower Bound} = \left( \frac{0.0001}{0.001} \right) \left( \frac{4}{6+1} \right) / F \left[ \frac{1+0.95}{2}; 2 \cdot 6 + 2, 2 \cdot 4 \right] = 0.014$$

$$\text{Upper Bound} = \left( \frac{0.0001}{0.001} \right) \left( \frac{4+1}{6} \right) \cdot F \left[ \frac{1+0.95}{2}; 2 \cdot 4 + 2, 2 \cdot 6 \right] = 0.281$$

In this example, because the upper bound of the ratio is  $< 1$ , it is concluded that Rate 1 (0.0004 s/cc) is less than Rate 2 (0.006 s/cc) at the 95% significance level.

### 3.0 REFERENCES

Nelson W. 1982. Applied Life Data Analysis. John Wiley & Sons, New York. pp 438-446.

# ATTACHMENT 5

## NVLAP Airborne Asbestos Proficiency Test 98-2: Grid Orientation

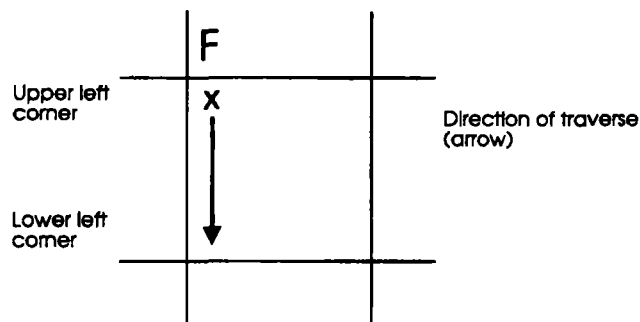
*This page intentionally left blank to facilitate double-sided printing.*

## NVLAP AIRBORNE ASBESTOS PROFICIENCY TEST 98-2

### *Instructions for Form 1*

The following procedure is designed to ensure that all laboratories count the grid squares in the same orientation and scan direction to allow for verified analyses which will be performed in the next round of proficiency testing.

1. Put a grid into the TEM. Find a particle at the magnification typically used for asbestos analysis. Move the particle using one stage translation and record the direction of movement of the particle on *Form 1*. Move the particle using the other stage translation knob and record the direction of movement. Recording the two directions of movement should roughly form a cross. The cross represents the translation directions of your microscope at the magnification used for asbestos analysis. ***Draw the letter "F" onto the cross so the sides of the letter are parallel to the translation directions and the letter is upright and is not inverted.*** See the example on *Form 1*.
2. Decrease the magnification and locate the letter "F" on the finder grid. Increase the magnification of the TEM to that typically used for asbestos analysis by your lab, keeping the letter "F" in the field of view. Compare the orientation of the "F" to the cross drawn in step 1. If the letter "F" is not oriented as shown in your sketch, remove the specimen holder and rotate or invert the grid as necessary to correctly align the grid. This may require several iterations.
3. When the correct orientation is found, record the grid's position in the specimen holder as shown in the example of the second part of *Form 1*. Indicate in your drawing where the straight side and the notched portion of the grid are located. All grids analyzed in this proficiency test should be oriented in the same manner (always check that the letter "F" is in the correct orientation and that the X-Y translation directions allow translation roughly parallel to the grid bars).
4. The starting point of the traverse for structure counting must correspond to the upper left corner on the grid square. The "X" marks the starting corner of the traverse (your grid square may be at an angle to that shown in the example):



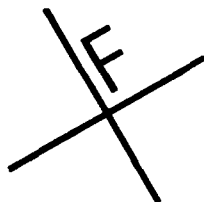
The initial direction of traverse must be from the upper left corner to the lower left corner of the grid square. If correctly oriented, the edge of the grid bar will remain in the field of view during the entire initial traverse (some allowance must be made for curvature or irregularly shaped grid bars.) If the grid is not oriented properly, go back to step 2.

NVLAP Lab Code: \_\_\_\_\_

**Form 1. Grid Orientation**

1. Sketch the orientation of the X-Y translation directions of the electron microscope as projected onto the electron microscope stage. Record the letter "F" as shown in the example below:

EXAMPLE:



2. Sketch below the orientation of the grid relative to the sample holder as shown in the example below:

EXAMPLE:



## ATTACHMENT 6

### Grid Opening Template for Sketching the Relative Position of Observed Structures



## STRUCTURE LOCATIONS WITHIN GRID OPENING

\*\*\*NOTE: Sketches only need to be completed for interlab analyses and reprep associated with interlabs

Lab Name: \_\_\_\_\_ Lab Job Number: \_\_\_\_\_

Index ID: \_\_\_\_\_ Lab Sample ID: \_\_\_\_\_

Lab QC Type (circle one):      Reprep for interlab      Interlab

Grid: \_\_\_\_\_ Grid Opening: \_\_\_\_\_

upper  
left  
corner

traverse direction

Comments:



## Request for Modification

To  
Laboratory Activities  
LB-000030

**Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.**

**File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:**

All Lab Applicable forms – copies to: EPA, Volpe, CDM-Denver, All project labs

Individual Lab Applicable forms – copies to: EPA, Volpe, CDM-Denver, Initiating Lab

Method (circle one/those applicable): TEM-AHERA, TEM-ISO 10312, PCM-NIOSH 7400, PLM-NIOSH 9002,  
EPA/600/R-93/116, ASTM D5755-95, EPA/540/2-90/005a, Other: EPA/600/R-94/134 (EPA 100.2)

Requester: W.J. Brattin

Title: Technical consultant

Company: Syracuse Research Corporation

Date: 5 August 2003

### Description of Modification:

All samples analyzed by TEM shall include sketches of all asbestos structures observed, up to a maximum of 50 structures in a sample. These sketches need not be highly detailed, but should include an indication of structure appearance and orientation relative to any nearby landmarks, if present.

morphology,

### Reason for Modification:

This modification is needed to standardize the procedure used by each laboratory for recording sketches of asbestos structures. One benefit of this modification is that samples for verified analysis no longer need to be identified before analysis.

### Potential Implications of this Modification:

There are no potential negative implications resulting from this standardization of QC procedures.

Laboratory Applicability (circle one): All

Individual: \_\_\_\_\_

### Duration of Modification (circle one):

Temporary

Date(s): \_\_\_\_\_

Analytical Batch ID: \_\_\_\_\_

Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

Permanent

(complete Proposed Modification Section)

Effective Date: 8/14/03 (insert based on date of final approval)

Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):  
\_\_\_\_\_  
\_\_\_\_\_

Technical Review: WJ Brattin

(Laboratory Manager or designate)

Date: 8/14/03

Project Review and Approval: Paul B.

(Volpe: Project Technical Lead or designate)

Date: 8/14/03

Approved By: Jaeey Coldade

(USEPA: Project Chemist or designate)

Date: 8/14/03

## Autio, Anni

**From:** Goldade.Mary@epamail.epa.gov  
**Sent:** Thursday, August 07, 2003 10:43 AM  
**To:** Autio, Anni  
**Cc:** Bob Shumate; Charlie LaCerra; Kyeong Corbin; Denise Mazzaferro; Gustavo Delgado; Garth Freeman; Jeanne Orr; Kwiatkowski, Joseph; Marie Cash; 'EMSL Mobile Lab - Asbestos'; ncbatta@battaenv.com; Mark Raney (raney@volpe.dot.gov); Rob DeMalo; Richard Hatfield; Ron Mahoney; Shu-Chun Su; Bill Longo  
**Subject:** EPA Comments: LB-000030 (Draft for review/comment)



LB-000030 v0 (MG pic08313.gif (3 KB)  
08-07-03).doc...

Attached are my recommended mark-ups. I also included Jeanne's recommendation of "if present" after landmarks. Please review and comment as nec.

One other point of clarification....when we discussed this, we were focused on AHERA. Just want to make sure it's OK w/ all to include TEM ISO on this list of circled methods. Thanks, Mary (See attached file: LB-000030 v0 (MG 08-07-03).doc) (Embedded image moved to file: pic08313.gif)



## Request for Modification

To  
Laboratory Activities  
LB-000030

**Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.**

**File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:**

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Individual Lab Applicable forms – copies to: EPA, Volpe, CDM-Denver, Initiating Lab

Method (circle one/those applicable): TEM-AHERA, TEM-ISO 10312, PCM-NIOSH 7400, PLM-NIOSH 9002,  
EPA/600/R-93/116, ASTM D5755-95, EPA/540/2-90/005a, Other: EPA/600/R-94/134 (EPA 100.2)

Requester: W.J. Brattin

Title: Technical consultant

Company: Syracuse Research Corporation

Date: 5 August 2003

### Description of Modification:

All samples analyzed by TEM shall include sketches of all asbestos structures observed, up to a maximum of 50 structures in a sample. These sketches need not be highly detailed, but should include an indication of structure appearance, morphology and orientation relative to any nearby landmarks, if present.

Deleted: \_\_\_\_\_

### Reason for Modification:

This modification is needed to standardize the procedure used by each laboratory for recording sketches of asbestos structures. One benefit of this modification is that samples for verified analysis no longer need to be identified before analysis and will be randomly selected by the laboratory's supervisor or designate following analysis.

### Potential Implications of this Modification:

There are no potential negative implications resulting from this standardization of QC procedures, but a benefit is that samples selected for verified analyses will be unknown to the microscopist prior to analysis.

Laboratory Applicability (circle one):

All

Individual: \_\_\_\_\_

Duration of Modification (circle one):

Temporary

Date(s): \_\_\_\_\_

Analytical Batch ID: \_\_\_\_\_

Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

Permanent

(complete Proposed Modification Section)

Effective Date: (insert based on date of final approval)

Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):

Deleted: \_\_\_\_\_

Technical Review: \_\_\_\_\_

(Laboratory Manager or designate)

Date: \_\_\_\_\_

Project Review and Approval: \_\_\_\_\_

(Volpe: Project Technical Lead or designate)

Date: \_\_\_\_\_

Approved By: \_\_\_\_\_

(USEPA: Project Chemist or designate)

Date: \_\_\_\_\_

**Autio, Anni**

**From:** DeMalo, Robert [RDemalo@EMSL.com]  
**Sent:** Thursday, August 07, 2003 11:20 AM  
**To:** Goldade.Mary@epamail.epa.gov; Autio, Anni  
**Cc:** Bob Shumate; LaCerra, Charles; Kyeong Corbin; Denise Mazzaferro; Gustavo Delgado; Garth Freeman; Jeanne Orr; Kwiatkowski, Joseph; Marie Cash; EMSL Mobile Lab - Asbestos; ncbatta@battaenv.com; Mark Raney (raney@volpe.dot.gov); Richard Hatfield; Mahoney, Ron; Shu-Chun Su; Bill Longo  
**Subject:** RE: EPA Comments: LB-000030 (Draft for review/comment)

I propose adding the word "morphology" as well into the description, as noted. I have no problem with including ISO to this procedure.

-----Original Message-----

**From:** Goldade.Mary@epamail.epa.gov [mailto:Goldade.Mary@epamail.epa.gov]  
**Sent:** Thursday, August 07, 2003 10:43 AM  
**To:** Autio, Anni  
**Cc:** Bob Shumate; Charlie LaCerra; Kyeong Corbin; Denise Mazzaferro; Gustavo Delgado; Garth Freeman; Jeanne Orr; Kwiatkowski, Joseph; Marie Cash; 'EMSL Mobile Lab - Asbestos'; ncbatta@battaenv.com; Mark Raney (raney@volpe.dot.gov); Rob DeMalo; Richard Hatfield; Ron Mahoney; Shu-Chun Su; Bill Longo  
**Subject:** EPA Comments: LB-000030 (Draft for review/comment)

Attached are my recommended mark-ups. I also included Jeanne's recommendation of "if present" after landmarks. Please review and comment as nec.

One other point of clarification....when we discussed this, we were focused on AHERA. Just want to make sure it's OK w/ all to include TEM ISO on this list of circled methods. Thanks, Mary (See attached file: LB-000030 v0 (MG 08-07-03).doc) (Embedded image moved to file: pic08313.gif)

**Autio, Anni**

**From:** Raney, Mark [RANEY@VOLPE.DOT.GOV]  
**Sent:** Thursday, August 14, 2003 10:41 AM  
**To:** 'Goldade.Mary@epamail.epa.gov'; Autio, Anni  
**Cc:** Bob Shumate; Charlie LaCerra; Kyeong Corbin; Denise Mazzaferro; Gustavo Delgado; Garth Freeman; Jeanne Orr; Kwiatkowski, Joseph; Marie Cash; 'EMSL Mobile Lab - Asbestos'; ncbatta@battaenv.com; Raney, Mark; Rob DeMalo; Richard Hatfield; Ron Mahoney; Shu-Chun Su; Bill Longo  
**Subject:** RE: EPA Comments: LB-000030 (Draft for review/comment)



LB-000030 v0 (MR  
08-14-03).doc...

I concur with Mary's recommendations and mark-ups. The attached version also includes Rob Demalo's recommendation of adding morphology under the description section. Bill please finalize, sign and send it through the signature process. To expedite the process could you get Mary to sign before providing the original on for my signature. Let me know if you have any questions.

Thanks,

Mark.

-----Original Message-----

**From:** Goldade.Mary@epamail.epa.gov [mailto:Goldade.Mary@epamail.epa.gov]  
**Sent:** Thursday, August 07, 2003 10:43 AM  
**To:** Autio, Anni  
**Cc:** Bob Shumate; Charlie LaCerra; Kyeong Corbin; Denise Mazzaferro; Gustavo Delgado; Garth Freeman; Jeanne Orr; Kwiatkowski, Joseph; Marie Cash; 'EMSL Mobile Lab - Asbestos'; ncbatta@battaenv.com; Mark Raney (raney@volpe.dot.gov); Rob DeMalo; Richard Hatfield; Ron Mahoney; Shu-Chun Su; Bill Longo  
**Subject:** EPA Comments: LB-000030 (Draft for review/comment)

Attached are my recommended mark-ups. I also included Jeanne's recommendation of "if present" after landmarks. Please review and comment as nec.

One other point of clarification....when we discussed this, we were focused on AHERA. Just want to make sure it's OK w/ all to include TEM ISO on this list of circled methods. Thanks, Mary (See attached file: LB-000030 v0 (MG 08-07-03).doc) (Embedded image moved to file: pic08313.gif)



## Request for Modification

To  
Laboratory Activities  
LB-000030

**Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.  
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Method (circle one/those applicable): TEM-AHERA, TEM-ISO 10312, PCM-NIOSH 7400, PLM-NIOSH 9002,  
EPA/600/R-93/116, ASTM D5755-95, EPA/540/2-90/005a, Other: EPA/600/R-94/134 (EPA 100.2)

Requester: W.J. Brattin Title: Technical consultant

Company: Syracuse Research Corporation Date: 5 August 2003

### Description of Modification:

All samples analyzed by TEM shall include sketches of all asbestos structures observed, up to a maximum of 50 structures in a sample. These sketches need not be highly detailed, but should include an indication of structure appearance, morphology and orientation relative to any nearby landmarks, if present.

Deleted: !

### Reason for Modification:

This modification is needed to standardize the procedure used by each laboratory for recording sketches of asbestos structures. One benefit of this modification is that samples for verified analysis no longer need to be identified before analysis and will be randomly selected by the laboratory's supervisor or designate following analysis.

### Potential Implications of this Modification:

There are no potential negative implications resulting from this standardization of QC procedures, but a benefit is that samples selected for verified analyses will be unknown to the microscopist prior to analysis.

Laboratory Applicability (circle one): All Individual: \_\_\_\_\_

### Duration of Modification (circle one):

Temporary

Date(s): \_\_\_\_\_

Analytical Batch ID: \_\_\_\_\_

Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

Permanent

(complete Proposed Modification Section)

Effective Date: (insert based on date of final approval) \_\_\_\_\_

Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable): \_\_\_\_\_

Deleted: !

Technical Review: \_\_\_\_\_ Date: \_\_\_\_\_  
(Laboratory Manager or designate)

Project Review and Approval: \_\_\_\_\_ Date: \_\_\_\_\_  
(Volpe: Project Technical Lead or designate)

Approved By: \_\_\_\_\_ Date: \_\_\_\_\_  
(USEPA: Project Chemist or designate)



**Request for Modification**  
to  
**Laboratory Activities**  
LB-000066c

**Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.**  
**File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:**

All Labs Applicable forms – copies to: EPA, Volpe, CDM, All project labs  
Individual Labs Applicable forms – copies to: EPA, Volpe, CDM, Initiating Lab

Method (circle one/those applicable): TEM-AHERA TEM-ISO 10312 PCM-NIOSH 7400 NIOSH 9002  
EPA/600/R-93/116 ASTM D5755 EPA/540/2-90/005a SRC-LIBBY-03  
Other: \_\_\_\_\_

Requester: W. Brattin Title: Technical Consultant  
Company: Syracuse Research Corporation Date: 09/11/2007

**Description of Modification:**

This temporary modification applies to all investigative samples (as defined by the most recent version of LB-000053) evaluated at the Libby Superfund site. Based on this temporary modification, all analytical laboratories shall: 1) begin to utilize the structure comment field to further characterize particles with regard to the levels (presence/absence) of the sodium and potassium peaks observed in the EDS spectrum; 2) record on the data sheets all NAM particles that are "close calls" (defined in attachment 1); 3) increase the frequency that EDS spectra are saved for "LA" and "close call" structures; 4) increase the frequency that photographic images of particle morphology are recorded for "LA" and "close call" structures, and 5) utilize the comment field to record mineral type of each recorded particle, including LA, OA, C and "close call" NAM particles.

**Reason for Modification:**

Studies of asbestos from the mine in Libby indicate that the asbestos spans several different mineralogical classes, including winchite and richterite (these are the primary forms) as well as tremolite and possibly actinolite (these are minor forms) (Meeker et al, 2003). Consequently, all analytical laboratories supporting the Libby project are currently directed to classify as "LA" any particle in an investigative sample that a) meets morphological requirements (e.g., length  $\geq 0.5$   $\mu$ m, aspect ratio  $\geq 3:1$ ), b) has an SAED diffraction pattern that is consistent with amphibole, and c) has an EDS spectrum that is consistent with the range of mineral forms observed in the mine in Libby (USEPA 2005). To date, this method for designating "LA" to a particle has worked well for samples collected at the Libby Site. However, a recent project that included collection of air samples from locations outside of Libby highlighted a potential limitation of this approach. That is, tremolite and actinolite are included in the "LA" suite and are found in Libby, but these types of fibers may also occur as the result of releases from sources that are not related to the mine in Libby (e.g., commercial products or natural sources). Also, some other minerals (e.g., pyroxenes) are sometimes difficult to distinguish from actinolite and tremolite (Bern et al. 2002). Because mineralogical data may or may not inform our understanding of the toxicity of LA, delineating amongst these mineral types is desirable at this stage of data collection. Therefore, the primary focus of this temporary modification is to collect more detailed data on the frequency of occurrence of sodium and potassium-containing particles both for samples from Libby and for samples from other locations.

**Potential Implications of this Modification:**

This temporary modification does not change any current procedures other than to require more detailed recording of data on particles observed under TEM. These additional requirements are not associated with a significant increase in time or cost of analysis. Hence, there are no negative implications of the modification.



Laboratory Applicability (circle one): All Individual(s) \_\_\_\_\_

Duration of Modification (circle one):

Temporary Date(s): 09/12/2007 until notified

Analytical Batch ID: \_\_\_\_\_

*Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages*

Permanent (Complete Proposed Modification Section) Effective Date: \_\_\_\_\_

*Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts*

Data Quality Indicator (circle one) – Please reference definitions on reverse side for direction on selecting data quality indicators:

Not Applicable

Reject

Low Bias

Estimate

High Bias

No Bias

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):

See Attachment 1

Note: This modification (LB-000066c) **supersedes** LB-000066b.

Technical Review: \_\_\_\_\_ Date: \_\_\_\_\_  
(Laboratory Manager or designate)

Project Review and Approval: [Signature] Date: 9/12/07  
(Vice Project Technical Lead or designate)

Approved By: [Signature] Date: 9/11/07  
(USEPA Project Chemist or designate)

## REFERENCES

Bern A, Meeker G, Brownfield I. 2002. Guide to Analysis of Soil samples from Libby, Montana for Asbestos Content by Scanning Electron Microscope and Energy Dispersive Spectroscopy. U. S. Geological Survey Administrative Report. October 17, 2002.

Meeker GP, Bern AM, Brownfield IK, Lowers HA, Sutley SJ, Hoeffen TM, and Vance JS. 2003. The Composition and Morphology of Amphiboles from the Rainy Creek Complex, Near Libby Montana. American Mineralogist 88:1955-1969.

USEPA. 2005. EDS Spectra Characteristic Study for Libby-Type Amphiboles. Report prepared by Syracuse Research Corporation, Denver CO, for USEPA, Region 8, Denver CO. March 15, 2005.

## DATA QUALITY INDICATOR DEFINITIONS

**Reject** - Samples associated with this modification form are not useable. The conditions outlined in the modification form adversely effect the associated sample to such a degree that the data are not reliable.

**Low Bias** - Samples associated with this modification form are useable, but results are likely to be biased low. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated low.

**Estimate** - Samples associated with this modification form are useable, but results should be considered approximations. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimates.

**High Bias** - Samples associated with this modification form are useable, but results are likely to be biased high. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated high.

**No Bias** - Samples associated with this modification form are useable as reported. The conditions outlined in the modification form suggest that associated sample data are reliable as reported.

## ATTACHMENT 1

1. Continue to classify structures as LA, OA, or C in accord with current procedures.
2. For all NAM particles that were "close calls" (i.e., they required careful assessment to determine they were not LA or OA), record the NAM particle on the bench sheet. Be sure to place a zero in the "total" column to ensure the particle is not counted as an asbestos fiber. NAM particles such as vermiculite, biotite, hydrobiotite, gypsum, titanium and other minerals that are clearly not amphibole should not be recorded.
3. For all particles that are recorded (including NAMs), use the structure comment field to record one of the following comments:

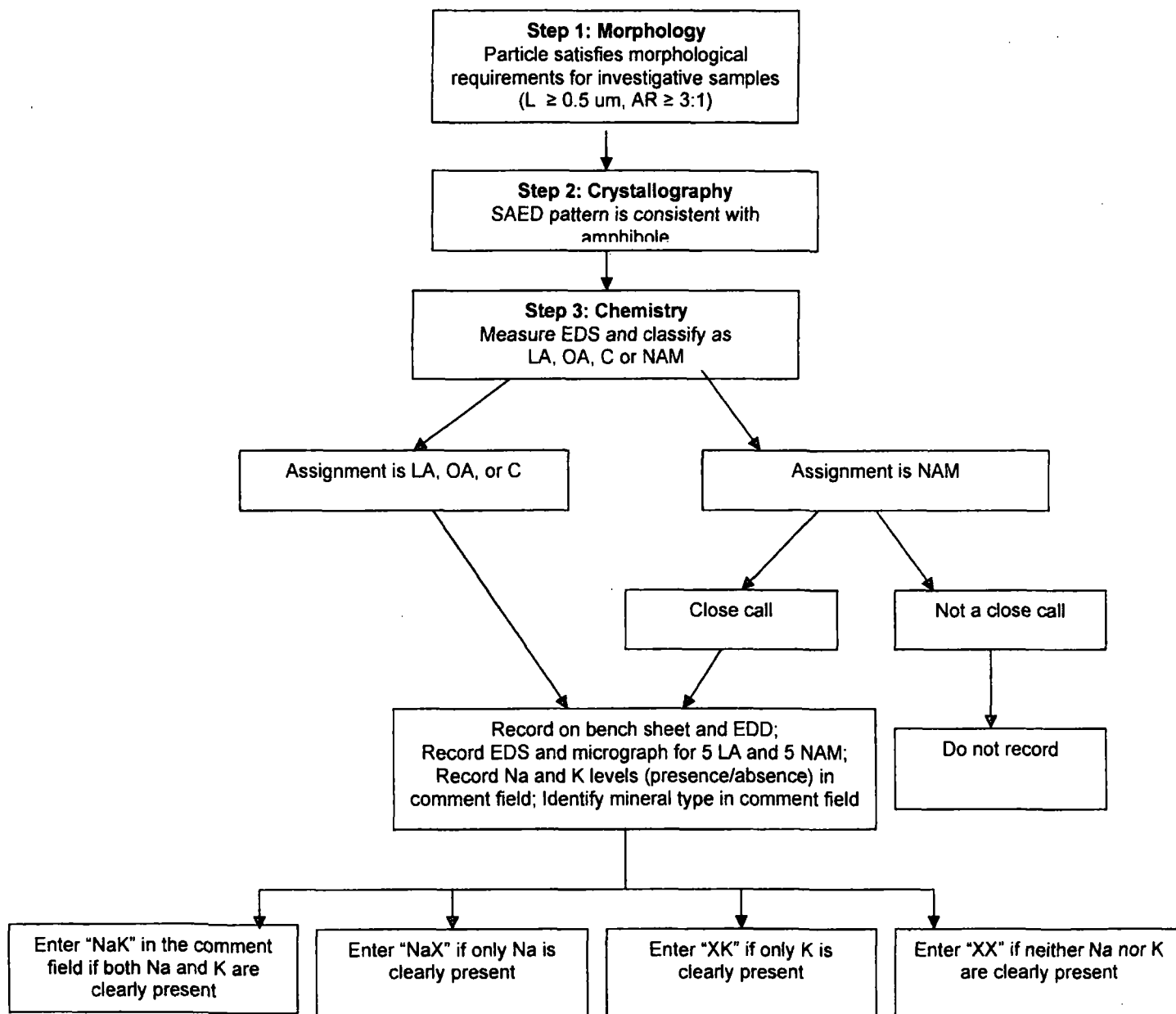
Code	Meaning
NaK	Na and K are both clearly present
NaX	Only Na is clearly present
XK	Only K is clearly present
XX	Na and K are not clearly present

4. For all particles that are recorded, whenever possible, use the structure comment field to identify a probable mineral classification. Use the designation "WRTA" (winchite/richterite/tremolite/actinolite) to indicate a particle that is consistent in morphology and chemical composition with a particle that is likely to have originated from the vermiculite mine in Libby. This will include most NaK particles and may include some NaX and some XK particles. It is unlikely that this will include any XX particles. For all other particles, use the following codes:

AC – actinolite  
TR – tremolite  
AT – actinolite/tremolite (too close to call)  
AM – amosite  
AN – anthophyllite  
CR – crocidolite  
PY – pyroxene  
UN – Unknown

5. Increase the frequency that EDS spectra are recorded (saved). For each sample, record the EDS for each LA and each "close call" particle, up to a maximum of 5 LA and 5 "close call" particles per sample. To the extent practical, collect the EDS spectrum for a sufficient length of time that key peaks (e.g., sodium, potassium, aluminum), if present, can be clearly distinguished from background. Be sure that each EDS spectrum that is recorded can be linked to a specific particle in the EDD.
6. Increase the frequency that photomicrographic images of particle morphology are collected. For each particle for which an EDS spectrum is collected (up to 5 LA and 5 "close call" NAM, as discussed above), also record a photomicrograph of the same structures. Use the structure-specific comment field to record the photo identification number of each structure that is photographed. Convert all photographs to high quality electronic images (e.g., by scanning), and transmit the photos to CDM for evaluation.
7. Figure 1 provides a flow chart that summarizes the process implemented by this temporary modification.

**FIGURE 1**  
**FLOW CHART SUMMARIZING THIS TEMPORARY MODIFICATION**





**Request for Modification**  
to  
**Laboratory Activities**  
LB-000085

**Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.**  
**File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:**

All Labs Applicable forms – copies to: EPA, Volpe, CDM, All project labs  
Individual Labs Applicable forms – copies to: EPA, Volpe, CDM, Initiating Lab

Method (circle one/those applicable): TEM-AHERA TEM-ISO 10312 PCM-NIOSH 7400 NIOSH 9002  
EPA/600/R-93/116 ASTM D5755 EPA/540/2-90/005a SRC-LIBBY-03  
Other: All TEM and SEM Methods supporting Libby site investigative or Libby Action Plan (LAP)  
sample analysis

Requester: Mary Goldade Title: Senior Environmental Scientist/Chemist  
Company: Environmental Protection Agency, Region 8 Date: April 2, 2008

**Description of Modification:**

Laboratories conducting transmission electron microscopy (TEM) or scanning electron microscopy (SEM) analysis in support of either the Libby Site (all operable units, including Troy) or Libby Action Plan shall perform analysis of a reference standard to calibrate the energy dispersive x-ray spectrometry (EDS) analysis. The reference standard, a glass material referred as BIR-1G, was created by the USGS. It is recommended for use for Libby Amphibole analysis because it contains sodium (Na) and potassium (K) at known levels. Na and K are important elements used in Libby Amphibole identification by EDS. The BIR-1G standard was freezer-milled by EMSL to create particles for EM analysis. While generation of thin sections of the BIR-1G using a microtome was not feasible due to the expense, analysis of the BIR-1G in particulate form is useful in standardizing the elemental measurements of the EDS and understanding the inherent variability in the EDS measurements.

The BIR-1G shall be tested daily (on days that the TEM scope is used for analysis of Libby samples) and must meet acceptance criteria prior to analysis of any field samples. Laboratories shall record the calibration information in accord with Attachment 1. As seen, not only does Attachment 1 provides the details for populating the electronic disk deliverable (EDD) used in recording the calibration information, but Attachment 1 also describes the process for generating acceptance criteria for the BIR-1G standard for each individual instrument.

**Reason for Modification:**

The modification provides for a standardized process for performing and recording calibration standards for EDS during Libby Amphibole analysis.

Potential Implications of this Modification: There are no negative implications to this modification. Positive impacts include a standardized process for: (1) daily calibration of a standard for the EDS used in Libby Amphibole identification; (2) reporting results of BIR-1G measurements; and (3) generating acceptance criteria for the BIR-1G standard over time.

Laboratory Applicability (circle one): All Individual(s) \_\_\_\_\_

This laboratory modification is (circle one): NEW APPENDS to \_\_\_\_\_ SUPERCEDES \_\_\_\_\_

**Duration of Modification (circle one):**

Temporary Date(s): \_\_\_\_\_  
Analytical Batch ID: \_\_\_\_\_

*Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages*

Permanent (Complete Proposed Modification Section) Effective Date: April 30, 2008  
*Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.*

Data Quality Indicator (circle one) – Please reference definitions on reverse side for direction on selecting data quality indicators:

Not Applicable

Reject

Low Bias

Estimate

High Bias

**No Bias**

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):

Technical Review: N/A Date: \_\_\_\_\_  
(Laboratory Manager or designate)

Project Review and Approval: \_\_\_\_\_ Date: \_\_\_\_\_  
(Volpe: Project Technical Lead or designate)

Approved By: \_\_\_\_\_ Date: \_\_\_\_\_  
(USEPA: Project Chemist or designate)

### DATA QUALITY INDICATOR DEFINITIONS

**Reject** - Samples associated with this modification form are not useable. The conditions outlined in the modification form adversely effect the associated sample to such a degree that the data are not reliable.

**Low Bias** - Samples associated with this modification form are useable, but results are likely to be biased low. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated low.

**Estimate** - Samples associated with this modification form are useable, but results should be considered approximations. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimates.

**High Bias** - Samples associated with this modification form are useable, but results are likely to be biased high. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated high.

**No Bias** - Samples associated with this modification form are useable as reported. The conditions outlined in the modification form suggest that associated sample data are reliable as reported.

## ATTACHMENT 1

**Analyzing the BIR-1G Standard**

- The BIR-1G standard shall be tested daily (on days that either the SEM or TEM microscope is used for analysis of Libby samples), prior to analyzing any field samples. Analyze for the compounds  $\text{Na}_2\text{O}$ ,  $\text{MgO}$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{SiO}_2$ ,  $\text{K}_2\text{O}$ ,  $\text{CaO}$ ,  $\text{TiO}_2$ ,  $\text{MnO}$ , and  $\text{FeO}$ . It is suggested that the reference publication for BIR-1G be reviewed. It is available in Volume 2 of the Analytical Guidance Documents, Tab 35, provided by CDM.
- Set up TEM instrument and orient for typical Libby field samples.
- Record the TEM instrument details in the BIR-1G Electronic Data Deliverable (EDD) spreadsheet (see most recent version of Excel file "BIR-1G EDD.xls"). Note: Use one spreadsheet per TEM instrument.
- For each daily BIR-1G evaluation, select one particle and record the measured weight % for each compound **as oxide weight %** in the BIR-1G EDD. Note: When recording oxide weight %, enter results as a percentage not fractions (i.e., for 30%, enter 30 not 0.3).
- When selecting particles for analysis:
  - Choose particles in the middle of the grid opening and in the center of the grid.
  - Particles should not be in close proximity to the grid bar or neighboring particles.
  - Randomly select particles within different grid openings for each analysis.
- For selected particles, focus the beam on the thin edge, not the center of the particle.
- Continue analysis until a maximum peak height count of at least 1,000 is achieved for silicon (Si). This total Si count should be sufficient to achieve optimum instrument testing conditions. It is recognized that this total Si count may not be equivalent to typical analytical conditions for field samples.
- On a monthly basis, the EDD for each TEM instrument should be provided to EPA (or designated contractors).

**Acceptance Criteria**

- Acceptance criteria will be TEM instrument- and element-specific and will be derived from measured results.
  - Results that are within  $\pm 1$  standard deviation of the nominal will be ranked as acceptable.
  - Results that are outside  $\pm 1$  standard deviation but within  $\pm 2$  standard deviations of the nominal will be ranked as within the warning level.
  - Results that are outside  $\pm 2$  standard deviations of the nominal will be ranked as a failure.
- The potential bias of measured results will be assessed based on a frequency evaluation of results above and below the nominal.
- As needed, EPA will re-evaluate and revise the acceptance criteria to optimize program goals.

**Corrective Action**

In the event that analysis results of the BIR-1G fall outside of the acceptance criteria, there should be a structured, progressive response. First, confirm that the detector/x-ray system has satisfied the acceptance criteria in the past. Next, confirm that the settings for the x-ray analysis software are correct (e.g. bias, scale). Finally, de-ice the LN2 dewar (unless it is a dry system) and carefully clean the window.

If these actions fail to rectify the problem, it will probably be necessary to send the detector/x-ray out to be serviced. The actions taken by the servicing company may include such things as baking the detector, renewing the vacuum in the dewar, checking the pre-amp or actual x-ray system for hardware defects, or replacing the crystal and/or FET (field effect transistor). In most instances the fault will not lie in the window unless the integrity of the window is compromised.

Upon the return and re-installation of the detector, re-run the BIR-1G standard to confirm that corrective action measures have resolved analysis issues.

**June 14, 2010**

**REMEDIAL INVESTIGATION  
FOR OPERABLE UNIT 3  
LIBBY ASBESTOS SUPERFUND SITE**

**CONTINGENCY AIR MONITORING PLAN  
DURING FOREST FIRES WITHIN OPERABLE UNIT 3**

**REVISION 1**

**Prepared by  
U.S. Environmental Protection Agency  
Region 8  
Denver, CO**



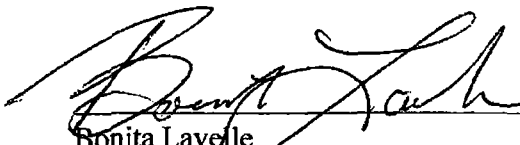
**With Technical Assistance from:**

**SRC, Inc.  
Denver, CO**



**APPROVAL PAGE**

The Contingency Air Monitoring Plan During Forest Fires Within Operable Unit 3 of the Libby Asbestos Superfund Site is approved for implementation.

  
\_\_\_\_\_  
Bonita Lavelle  
Remedial Project Manager, Libby OU3

6/14/10  
Date



### DOCUMENT REVISION LOG

Revision	Date	Primary Changes
0	08/11/09	--
1	06/14/10	Add mobile monitor downwind of fire

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## LIST OF FIGURES

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Figure 2 Windrose for Zonolite Mountain, Libby, MT

## LIST OF ATTACHMENTS

- Attachment 1 Libby-Specific Standard Operating Procedures (SOPs)  
Attachment 2 Field Sample Data Sheet (FSDS) Form  
Attachment 3 Volume Calculator Spreadsheet Tool (*provided electronically*)

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## **1.0 INTRODUCTION**

Studies performed to date as part of the Superfund Remedial Investigation (RI) for Operable Unit 3 (OU3) of the Libby Asbestos Superfund Site have revealed that soil, tree-bark, and duff in the vicinity of the former vermiculite mine have been impacted by historic releases of Libby amphibole asbestos (LA) (EPA 2008, EPA 2009). Forest fires that occur within contaminated areas of OU3 may result in the release of LA fibers into air although the magnitude of the release is unknown.

At present, no data are available on the concentration of LA fibers that may be released during fires in OU3. The purpose of this document is to present a plan for establishing air monitoring stations and for collecting air samples that will provide preliminary information on the levels of LA in ambient area that may occur in residential areas during forest fires in OU3 within the area designated as a Fire Suppression Restricted Zone (FSRZ) by the U.S. Forest Service (see yellow line in Figure 1). The resulting data may be useful in the RI for OU3 but the primary purpose of the data are to inform the general public and the U.S. Forest Service of air impacts from forest fires within OU3.

## **2.0 PROJECT MANAGEMENT AND ORGANIZATION**

EPA is the lead regulatory agency for Superfund activities within OU3. The EPA Remedial Project Manager (RPM) for OU3 is Bonita Lavelle, EPA Region 8. Ms. Lavelle is a principal data user and decision-maker for Superfund activities within OU3.

The Montana Department of Environmental Quality (MDEQ) is the support regulatory agency for Superfund activities within OU3. The MDEQ Project Manager for OU3 is Dick Sloan. EPA will consult with MDEQ regarding all Superfund investigations and assessments within OU3, as provided for by the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA), the National Contingency Plan, and other applicable guidance.

EPA has entered into an Administrative Order on Consent (AOC) with Respondents W.R. Grace & Co.-Conn. and Kootenai Development Corporation (KDC) to perform a Remedial Investigation/Feasibility Study (RI/FS) within OU3. Although the data to be collected under this plan are not directly in support of the RI/FS for OU3, W.R. Grace & Co.-Conn. and KDC have agreed to implement this SAP. The designated Project Coordinator for Respondents W.R. Grace & Co.-Conn. and KDC is Robert Medler of Remedium Group, Inc.

### 3.0 DATA QUALITY OBJECTIVES

The key data quality objective for air samples collected under this project is to collect data that may be used to estimate exposure to residents and workers in Libby in the event that a wildfire in OU3 generates significant levels of smoke in the community. This objective will be achieved by collecting two types of data:

1. Stationary air samples will be collected in and about the community of Libby when smoke from a fire in OU3 is reaching the community. This type of data provides a direct measure of human exposure to LA in smoke. However, collection of these data is contingent upon the occurrence of a fire in OU3 that generates smoke that reaches the community.
2. Stationary air samples will be collected downwind of the fire (regardless of the direction that smoke is blowing). These data are valuable because the measured levels of LA in smoke can be used to model (predict) the levels of LA that would occur in Libby if the smoke were to be blown toward the community.

All samples collected as part of this program will be analyzed with an analytical detection limit that is sufficient to allow reliable detection and quantification of LA fibers if they were present at a level of concern in air. The basic equation for calculating the concentration of potential concern is as follows (USEPA 2008):

$$RBC = \frac{\text{Target Risk}}{TWF \cdot IUR_{a,d}}$$

where:

RBC = Risk-based concentration (PCM f/cc)

TWF = time weighting factor to account for less than continuous exposure

IUR<sub>a,d</sub> = Inhalation unit risk for exposures that begin at age a and last for d years (PCM f/cc)<sup>-1</sup>

In order to be conservative, the following assumptions were used:

Target Risk = 1E-05

TWF = 2 days/fire, 5 fires/year = (240 hrs/yr) / (8760 hr/yr) = 0.027

a (age at first exposure) = 0 years

d (duration of residency in Libby) = 40 years

IUR<sub>0,40</sub> = 0.194 (PCM f/cc)<sup>-1</sup> (USEPA 2008)

Based on these conservative assumptions, the RBC equals about 0.002 PCM f/cc. Assuming that about 50% of all LA fibers are PCME, this corresponds to an RBC of about 0.004 LA f/cc. In order to ensure that the target sensitivity is adequate to ensure that samples with a true

concentration equal to the RBC can be accurately quantified, the target sensitivity is calculated as follows:

$$\text{Target sensitivity (cc)}^{-1} = \text{RBC (LA f/cc)} / 5 \text{ LA fibers} = 0.0008 \text{ (cc)}^{-1}$$

Based on this, the target sensitivity is set to  $0.0008 \text{ cc}^{-1}$ . This ensures that an average of 5 LA fibers would be detected if air levels during a fire reached or exceeded the RBC.

## **4.0 SAMPLING PLAN**

### **4.1 Sampling Station Locations**

During a fire in OU3, air monitoring will be performed at three fixed stations and one mobile station, as follows:

**Fixed Station 1:** Based on meteorological data collected at the mine site, the predominant wind direction at OU3 is to the north-northeast (see Figure 2). This means that smoke and LA released from fires in OU3 is most likely to be transported in that direction. Under current conditions, most of the land north and east of the former mine is owned by the U.S. Forest Service or by logging companies, and human occupancy in this area is sparse. Based on this, during a fire event, one monitoring station will be established at a location in the downwind direction, west of Lake Koocanusa within the camping area at McGillivray Access.

**Fixed Station 2:** Because Libby is the location of the highest population density near the mine, a second air monitor will be established on the east side of the town of Libby to provide information on exposure levels to this population. The location of this monitor will be at the CDM offices.

**Fixed Station 3:** A third monitoring station will be established along Highway 37 at the U.S. Forest Service Canoe Gulch Ranger Station. This location was chosen based on its proximity to OU3 and the fact that people routinely occupy the station during work hours.

**Mobile Station:** In addition to these three stationary monitors at fixed locations, a fourth monitor will be deployed to an area downwind of the fire. The monitor will be transported to the collection site by truck. The sampling location and distance from the fire will depend on the conditions of the fire. Although details may vary, it is envisioned that the monitor will be placed on a tripod in the back of the truck. During sample collection, the coordinates of the monitor will be recorded. This information will be used later, in combination with data on the fire location, to establish the distance and direction of the monitor relative to the fire. The wind direction and speed at the sampling location should also be monitored.

## 4.2 Sample Collection Schedule

Air sampling at the three fixed monitoring stations will not occur except during times that a fire is burning in or near the FSRZ of OU3, and smoke from the fire is reaching the vicinity of one or more of the fixed monitors. [Note: This may include any controlled burns conducted by the USFS and the setting of "simulated wildfires" as part of EPA's Phase 4 Remedial Investigation, as may be appropriate.]

Notification that a fire is occurring in or near the FSRZ in OU3 will be provided to the field sampling team by the Forest Service as soon as possible after a fire is known to be occurring. If smoke is blowing toward Libby, the field crews will then activate all three monitors as soon as possible after notification. The person to be contacted in the event of a fire within the FSRZ is:

Mike Chapman  
406-293-1983  
[chapman@montanasky.net](mailto:chapman@montanasky.net)

## 4.3 Sample Collection Protocol

All air samples will be collected in basic accord with SOP EPA-LIBBY-01 (Rev. 1, March 2001) (see Attachment 1). Each air sample will collected using a stationary air monitor.

Pumps may be either battery-powered or provided with 110 volt power from a reliable source. Air sampling cassettes will utilize a 25 mm diameter mixed cellulose ester (MCE) with a pore size of 0.8 um. Target pump rates will be  $5 \pm 0.5$  L/min.

Each air sampling pump will be calibrated at the start of each sampling event using a rotameter that has been calibrated to a primary calibration source. Calibration will be considered complete when the measured flow is within  $\pm 10\%$  of the target flow (5 L/min), as determined by the mean of three measurements.

For the three fixed air monitoring stations, each sample will be collected over a time period of about 24 hours. Sample collection will be repeated for 24-hour intervals as long as smoke from the fire continues to reach the community.

For the mobile air monitor, the sampling time depends on the level of smoke reaching the sampling station, as well as on the speed that the fire is moving. Assuming that there are no safety concerns, the sampling duration for the mobile monitor shall be about 30-60 minutes, depending on smoke level.

**NOTE: In all cases, it is critical that mobile station sampling be performed in a way that does not endanger that health or safety of the sampling personnel. If conditions are considered to be potentially unsafe, the sampler should evacuate the area immediately.**

## 4.4 Sample Documentation and Handling

### Sample Documentation

Data regarding each sample collected as part of monitoring activities will be documented using a Libby OU3-specific field sample data sheet (FSDS) designed specifically for this plan (see Attachment 2). At the time of collection, each sample will be labeled with a unique 5-digit sequential identification (ID) number. The sample IDs for all samples collected as part of this monitoring plan will have a prefix of "SM" (e.g., SM-12345), unless specified otherwise. Information on whether the sample is representative of a field sample or a field-based QC sample (e.g., field blank) will be documented on the FSDS.

GPS coordinates for all sampling locations will be recorded on the FSDS form.

Each field sampling team will also maintain a field log book. The log book shall record all potentially relevant information on sampling activities and conditions that are not otherwise captured on the FSDS form. Examples of the type of information to be captured in the field log include:

- Names of team members
- Weather conditions
- Field sketches
- Physical description of the location relative to permanent landmarks
- Number and type of samples collected
- Any special circumstances that influenced sample collection
- Any deviations from sampling SOPs

### Chain of Custody and Shipment

A chain-of-custody (COC) form specific to the Libby OU3 sampling shall accompany every shipment of samples to the analytical laboratory. The purposes of the COC form are: a) to establish the documentation necessary to trace possession from the time of collection to final disposal, and b) to identify the type of analysis requested.

OU3-specific COC forms can be obtained from the MWH Field Office in Libby, MT (an example of this form is provided in OU3 SOP No. 9 *Field Documentation* [see Attachment B of the main report]). Air volume for each sample should be calculated using the "Volume Calculator.xls" spreadsheet tool (provided as Attachment 3) and recorded on the COC form. All corrections to the COC form will be initialed and dated by the person making the corrections. Each COC form will include signatures of the appropriate individuals indicated on the form. The originals will accompany the samples to the laboratory and copies documenting each custody



change will be recorded and kept on file. One copy of the COC form will be kept by field personnel.

All required paper work, including sample container labels, COC forms, custody seals and shipping forms will be fully completed in indelible ink (or printed from a computer) prior to shipping of the samples to the laboratory. Shipping to the appropriate laboratory from the field will occur through overnight delivery.

All samples that may require special handling by laboratory personnel to prevent potential exposure to LA or other hazardous substances will be clearly labeled.

## **5.0 ANALYSIS REQUIREMENTS**

All analytical laboratories that analyze samples of air for asbestos as part of this project must participate in and have satisfied the certification requirements in the last two proficiency examinations from the National Institute of Standards and Technology/National Voluntary Laboratory Accreditation Program (NVLAP).

All air samples collected during fires in OU3 will be analyzed using two methods.

### **5.1 PCM Analysis**

All air samples from the three fixed-based monitors and the downwind mobile monitor will be analyzed initially by phase contrast microscopy (PCM) in basic accord with NIOSH Method 7400. It should be noted that PCM is not specific for LA or asbestos, and results from PCM analyses may not be a reliable indicator of actual LA levels in air. However, because analysis by PCM is rapid, this method can provide initial results much faster than TEM, and PCM results may be sufficient to provide a basis for initial risk management decision-making until more reliable data can be obtained.

Assuming that the typical sample volume for a fixed-based air sample will be about 7,200 L (24 hours x 60 min/h x 5 L/minute), and that 100 fields of view (each 0.00785 mm<sup>2</sup>) are analyzed, this will achieve an analytical sensitivity of about 0.00007 cc<sup>-1</sup>, which exceeds the DQO described above.

### **5.2 TEM Analysis**

#### **Counting Rules**

Based on the results of PCM analysis, samples of air collected from stationary and mobile monitors may also be submitted for asbestos analysis using transmission electron microscopy (TEM) in basic accord with the International Organization for Standardization (ISO) 10312

method (ISO 1995) counting protocols, with all applicable Libby site-specific laboratory modifications, including the most recent versions of modifications LB-000016, LB-000019, LB-000028, LB-000030, LB-000066, and LB-000085 (see Attachment C of the main report). All structures that have Selective Area Electron Diffraction (SAED) patterns and Energy Dispersive X-Ray Analysis (EDXA) spectra that are consistent with LA, and have a length greater than or equal to 0.5  $\mu\text{m}$  and an aspect ratio (length:width)  $\geq 3:1$ , will be recorded on the Libby site-specific laboratory bench sheets and electronic data deliverable (EDD) spreadsheets. Data recording for chrysotile, if observed, is not required.

### Stopping Rules

For field samples, evaluate each sample until one of the following is achieved:

- A minimum of 2 grid openings (GOs) in each of 2 grids has been examined.
- A target sensitivity of 0.0008  $\text{cc}^{-1}$  is achieved. Assuming that the typical sample volume for a fixed-based sample will be about 7,200 L (24 hours x 60 min/h x 5 L/minute), that the sample may be analyzed with using a direct preparation, and that the area of a GO is 0.01  $\text{mm}^2$ , it is expected that an analytical sensitivity of 0.0008  $\text{cc}^{-1}$  can be achieved by counting about 8 GOs.
- 50 LA structures are observed
- An area of 0.5  $\text{mm}^2$  has been examined (approximately 50 GOs)

When one of these goals is achieved, complete the final GO and stop.

For lot blanks and field blanks, evaluate an area of 0.1  $\text{mm}^2$  (approximately 10 GOs) and stop.

## **5.3 Sample Archival and Final Disposition**

All sample materials, including filters, grids, and cassettes will be maintained in storage at the laboratory unless otherwise directed by EPA. When authorized by EPA, the laboratory will be responsible for proper disposal of any remaining samples, sample containers, shipping containers, and packing materials in accordance with sound environmental practice, based on the sample analytical results.

## **5.4 Quality Control**

### Field-Based Quality Control Samples

#### *Lot Blanks*

Before any air cassettes may be used for asbestos sampling, the lot must be determined to be asbestos free. This will be accomplished by selecting 2 lot blanks at random from the group of

cassettes to be used for collection of air samples. Each lot blank will be submitted for TEM analysis as described above. Once the lot is confirmed to be asbestos free (i.e., both lot blanks are non-detect after evaluation of an area of 0.1 mm<sup>2</sup>), that lot may be placed into use for sampling.

### *Field Blanks*

A field blank for air shall be prepared by removing the sampling cassette from the box, opening the cassette to the air in the area where the investigative samples will be taken, then closing the cassette and packaging for shipment and analysis. Field blanks will be collected at a rate of 1 per 2 days that sampling is occurring.

### *Field Duplicate*

A field duplicate air sample shall be collected at the downwind mobile monitoring station at the rate of 1 field duplicate per sampling event.

### Laboratory-Based Quality Control Samples

The laboratory QC requirements for analyses of air samples at the Libby site are patterned after the requirements set forth by NVLAP. There are three types of laboratory-based QC analyses that are performed. Each of these is described below:

*Lab Blank* - This is an analysis of a filter that is prepared from a new, unused filter in the laboratory and is analyzed using the same procedure as used for field blank samples.

*Recount* - A recount is an analysis where TEM grid openings are re-examined after the initial examination. For PCM, a *Blind Recount* (RB) is a slide that has been analyzed is re-labeled and re-submitted (blind) for a second analysis within the same laboratory. For TEM, a *Recount Different* (RD) describes a re-examination by a different microscopist within the same laboratory than who performed the initial examination.

*Repreparation* - A repreparation is an analysis of a TEM grid that is prepared from a new section of filter as was used to prepare the original grid(s). Typically, this is done within the same laboratory as did the original analysis, but a different laboratory may also prepare grids from a new piece of filter.

For this project, the frequency of each of these laboratory-based QC samples will be 1 per fire event (i.e., 1 lab blank, 1 recount, 1 repreparation per fire event). Samples for recount and repreparation will be selected by the analytical laboratory in accord with the selection procedures described in the most recent version of Libby laboratory modification #LB-000029.

## **6.0 DATA REPORTING REQUIREMENTS**

### **6.1 Reporting of Sample Information**

In the field, sample details and COC information will be documented on hard copy FSDS forms, field log books, and COC forms. Copies of all FSDS forms, field log books, and COC forms will be scanned and posted in portable document format (PDF) to a project-specific file transfer protocol (FTP) site at the end of each monitoring event. This FTP site will have controlled access (i.e., user name and password are required) to ensure data access is limited to appropriate project-related personnel. File names for scanned documents will include the sample date in the format MMDDYY to facilitate document organization (e.g., "FSDS\_083109.pdf").

A copy of the completed "Volume Calculator.xls" spreadsheet tool should also be posted to the FTP site at the end of each monitoring event. The file name for posted spreadsheets will include the date the file was posted to facilitate file tracking (e.g., "Volume Calculator\_083109.xls").

### **6.2 Reporting of Analytical Results**

All analytical results from air monitoring during fires will be provided to EPA for review and evaluation.

PCM results will be reported to EPA within 4 hours of sample receipt by the laboratory. The purpose of this rapid turn-around requirement is to allow EPA and the Forest Service to recognize if significant LA exposures are occurring in residential areas. The PCM data will be submitted to the EPA OU3 Project Manager electronically via email ([lavelle.bonita@epa.gov](mailto:lavelle.bonita@epa.gov)). The email shall be copied to Remedium at the email address [Robert.r.marriam@grace.com](mailto:Robert.r.marriam@grace.com). TEM results will be reported within one week of sample receipt by the laboratory. All TEM results will be submitted using the most recent version of the TEM EDD for air samples in use at the Libby site.

All TEM EDDs and scanned copies of all hard copy lab reports will be submitted to EPA's technical contractor (SRC) electronically. Whenever possible, data files should be transmitted by e-mail to the following address:

[LibbyOU3@srcinc.com](mailto:LibbyOU3@srcinc.com)

When files are too large to transmit by e-mail, they should be provided on compact disk to the following address:

Lynn Woodbury  
SRC, Inc.  
999 18th Street, Suite 1975  
Denver CO 80202  
(303) 357-3127

All original data records (both hard copy and electronic) will be cataloged and stored in their original form until otherwise directed by EPA.

### **6.3 Reports to Management**

Field and analytical staff will promptly communicate any difficulties or problems in implementation of this to EPA, and may recommend changes as needed. All recommendations should be documented in a memo to the EPA RPM for OU3 at the following address:

Bonita Lavelle  
U.S. EPA, Region 8  
1595 Wynkoop Street  
Denver, CO 80202-1129  
E-mail: [lavelle.bonita@epa.gov](mailto:lavelle.bonita@epa.gov)

If any revisions to this plan are needed, the EPA RPM will approve these revisions before implementation by field or analytical staff.

### **7.0 REFERENCES**

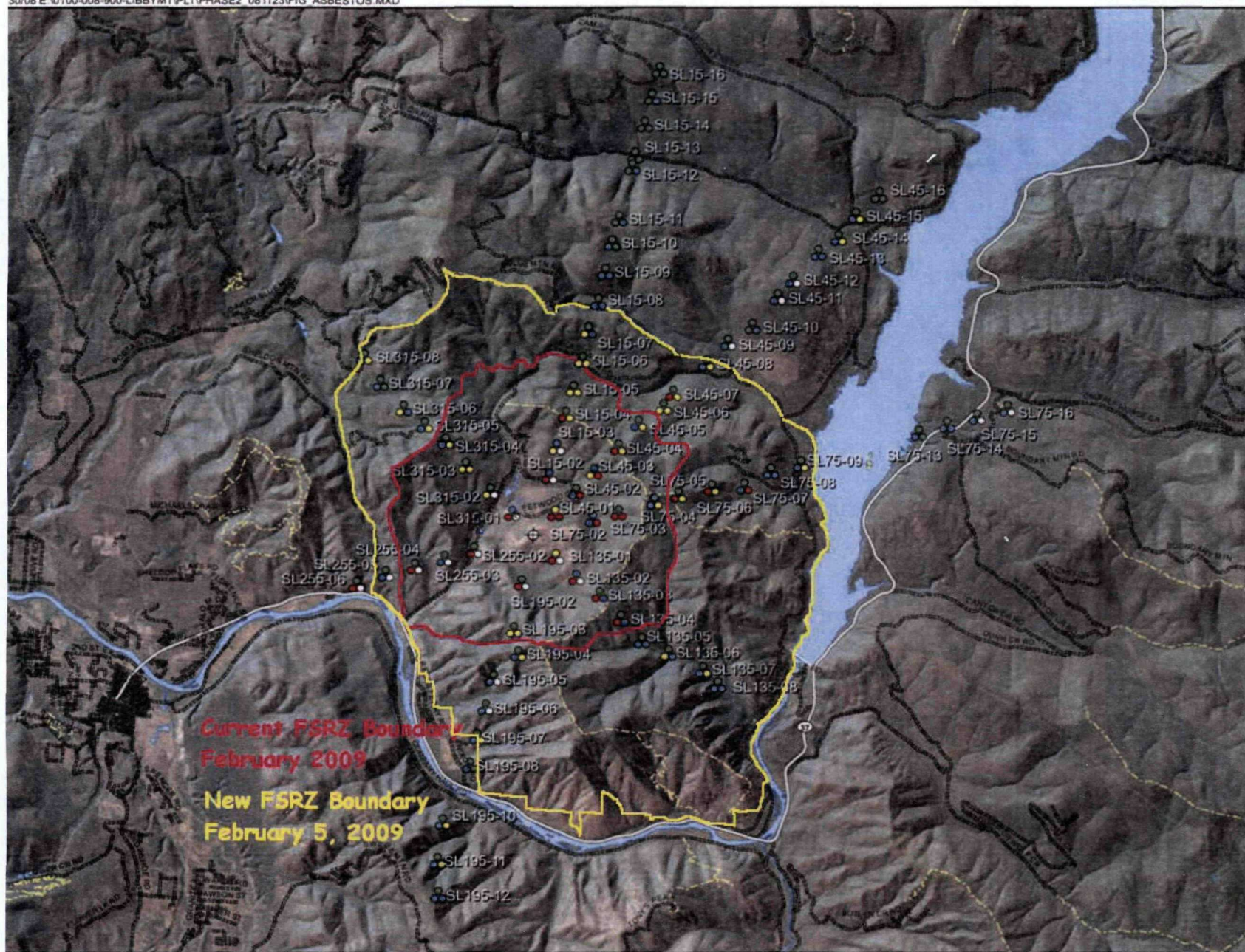
EPA. 2008. Phase II Sampling and Analysis Plan for Operable Unit 3 Libby Asbestos Site, Part C: Ecological Data. U.S. Environmental Protection Agency, Region 8. September 17, 2008.

EPA. 2009. Remedial Investigation for Operable Unit 3 Libby Asbestos Superfund Site Phase III Sampling and Analysis Plan. U.S. Environmental Protection Agency, Region 8. May 26, 2009.

## FIGURES

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### Legend

#### Asbestos in Tree Bark (LA Loading)

- ND
- < 1.0 MS/cm<sup>2</sup>
- 1.0 - 2.5 MS/cm<sup>2</sup>
- > 2.5 MS/cm<sup>2</sup>

#### Asbestos in Forest Soil (MFLA% fine)

- ND
- TR
- < 1%
- 2 - 7%

#### Asbestos in Duff (MFLA% Dried)

- ND
- < 0.01%
- 0.01 - 0.5%
- > 0.5%
- No Data

⊕ Origin of Transects

--- County Road

— Primary Road

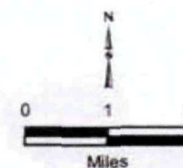
--- National Forest Service Trails

■ Open Water

#### Symbol Placement

Forest Soil

Tree Bark ● Duff



**LIBBY MONTANA SUPERFUND SITE**  
OPERABLE UNIT 3

FIGURE 4-10  
**ASBESTOS CONCENTRATIONS IN  
FOREST SOIL, TREE BARK,  
AND DUFF**

PROJECT: 0100-008-900 NOV 30, 2008

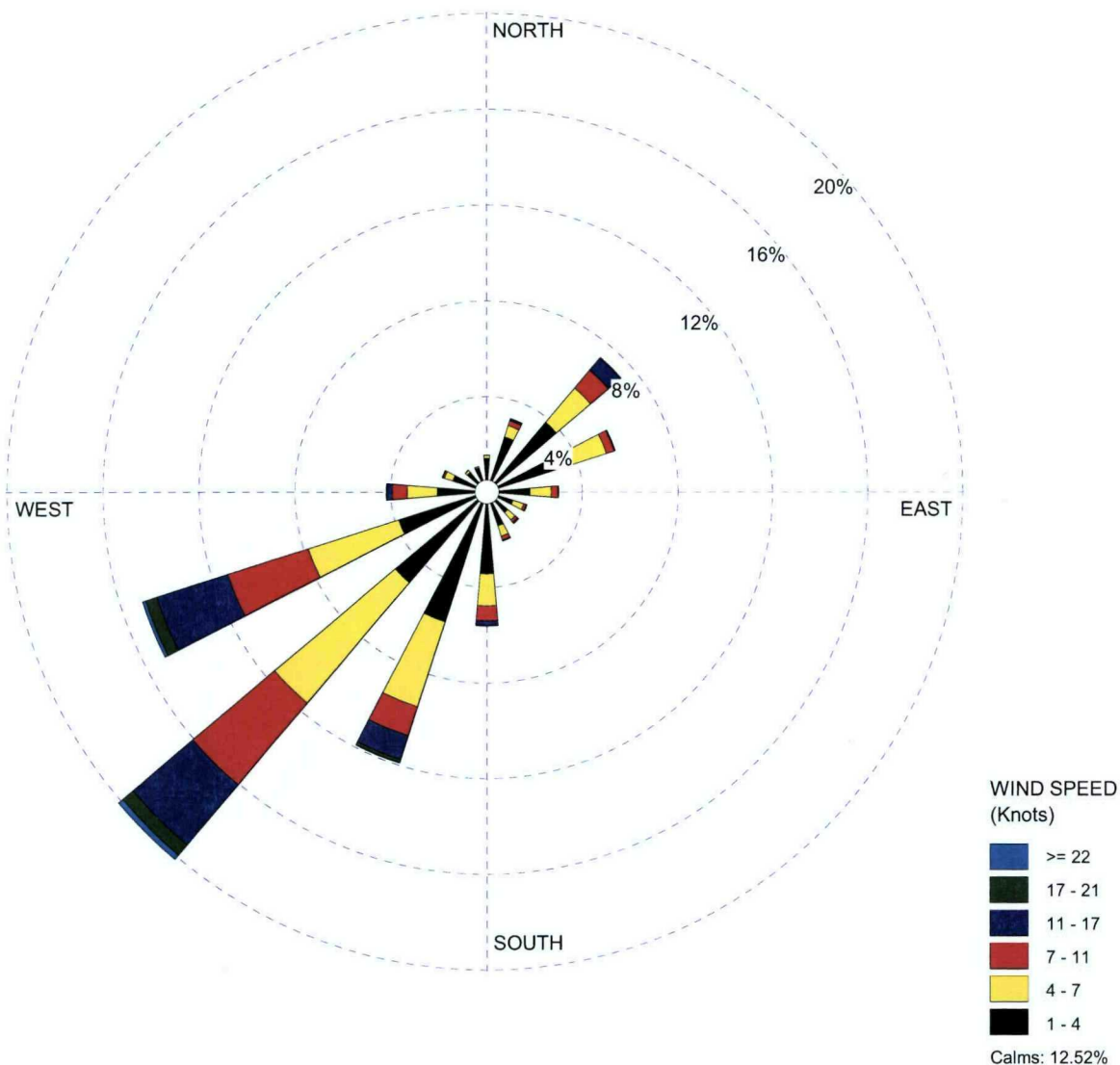
REV: 0 BY: VFS CHK: ACK

NEWFIELDS



**FIGURE 2**  
**WindRose for Zonolite Mountain, Libby, MT**

DISPLAY:  
**Wind Speed**  
**Direction (blowing from)**



COMMENTS:

DATA PERIOD:

**2006-2009**  
**Jan 1 - Dec 31**  
**00:00 - 23:00**

COMPANY NAME:

**W.R. Grace**

MODELER:

**MWH Global**



**MWH**

BUILDING A BETTER WORLD

CALM WINDS:

**12.52%**

TOTAL COUNT:

**26272 hrs.**

AVG. WIND SPEED:

**4.93 Knots**

DATE:

**2/26/2010**

PROJECT NO.:

**ATTACHMENT 1**

**LIBBY-SPECIFIC  
STANDARD OPERATING PROCEDURES (SOPs)**

SOP EPA-LIBBY-01 (Revision 1, March 2001)

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U.S. ENVIRONMENTAL PROTECTION AGENCY  
REGION 8

STANDARD OPERATING PROCEDURE (SOP)  
FOR THE SAMPLING OF ASBESTOS FIBERS IN AIR

Prepared by: *William D. Smith for W. Brattin*  
(Author)

Date: 3/8/01

Reviewed by: *Chloris*  
(Project Director)

Date: 3/8/01

*Wang Goldacke*  
(Quality Assurance Coordinator)

Date: 3/8/01

Approved by: *Chloris for Paul Perreand*  
(Project Manager)

Date: 3/9/01

## REVISION LOG

Revision Date	Reason for Revision
02/28/01	--
03/07/01	Further define pump calibration procedures.

## PROCEDURAL SECTION

### 1.0 Scope and Applicability

This Standard Operating Procedure (SOP) provides a standardized method for sampling air to measure the concentration of asbestos fibers. This SOP is applicable to any type of asbestos fiber (amphibole, chrysotile) that may exist in air (either indoor or outdoor), and is applicable to both personal and ambient air (referred as stationary air throughout this SOP) sampling techniques. Filters collected in this way are suitable for examination by a variety of microscopic techniques, including TEM, PCM, and SEM.

### 2.0 Summary of Method

This SOP is based on air sampling techniques described in EPA SOP 2015, ISO 10312, OSHA Technical Manual, NIOSH 7400 and NIOSH 7402.

Air is drawn through a fine-pore filter in order to trap any suspended particulate matter in the air, including suspended asbestos fibers and other mineralogic materials. The filters are then examined using an appropriate microscopic technique to observe, characterize and quantify the number of asbestos fibers on the filter. The concentration of fibers in air is then calculated by dividing the total number of fibers on the filter by the volume of air drawn through the filter.

### 3.0 Health and Safety Warnings

Asbestos fibers are hazardous to human health when inhaled. Exposure to excessive levels may increase the risk of lung cancer, mesothelioma, and asbestosis. All personnel engaged in collection of air samples in areas where asbestos fibers may be present must have adequate health and safety training and must wear an appropriate level of personal protective equipment (PPE). Refer to the Health and Safety Plan for further details.

### 4.0 Cautions

None, refer to Section 3.0.

### 5.0 Interferences

High levels of dust or other suspended particulates may clog or overload the filter and reduce the ability to observe and characterize asbestos fibers on the filters. Precautions should be taken to avoid any unnecessary sources of dust emissions or use of aerosol sprays. Sampling conditions

(flow rate, sampling time) should be adjusted accordingly to avoid filter overload.

## 6.0 Personnel Qualifications

Field personnel engaged in collection of filter cassettes must be trained in the proper use and calibration of the air sampling equipment (as specified in this SOP), as well as proper methods for data recording and sample handling. Additionally, all field personnel must maintain appropriate and current training and/or certifications to meet all federal, state, and local regulations.

## 7.0 Apparatus and Equipment

### Filter Cassettes

All samples will be collected on conductive filter holders consisting of 25-mm diameter, three piece filter cassettes having a 50-mm long electrically conductive extension cowl. The cassette shall be pre-loaded with a mixed cellulose ester (MCE) filter with pore size 0.8  $\mu\text{m}$ . Use of the 0.8  $\mu\text{m}$  pore size is recommended for all samples so that samples collected using a high volume pump are comparable to samples collected with a low volume pump. The 0.8  $\mu\text{m}$  pore size filters are used for samples collected with a low volume pump in order to decrease back-pressure and increase flow rate.

To reduce contamination and to hold the cassette tightly together, seal the crease between the cassette base and the cowl with a shrink band or adhesive tape. If particle deposition on the inside of the cowl is observed, it may be necessary to ground the cowl to reduce static charge. This is done by attaching one end of a length of flexible wire to the plastic cowl with a hose clamp and attaching the other end of the wire to a suitable ground (e.g., a cold water pipe).

### Air Pumps

The sampling pump used shall provide a non-fluctuating airflow through the filter and shall maintain the initial flow rate within  $\pm 10\%$  throughout the sampling period.

A variety of different types of air pump may be used, depending on the flow rates that are required to achieve the data quality objectives and desired analytical sensitivity of the project. In general, the pump should be selected to deliver a flow rate that is as high as possible without overloading the filter with dust or fibers. The minimum flow rate is 0.5 L/min, and rates up to 10 L/min may be appropriate in some cases.

For stationary air monitors, a high volume pump that operates on AC power is recommended. For personal air sampling, either a portable high volume AC powered sampler or a low volume

battery-operated pump are acceptable, depending on whether the activities of the individual are impaired by the tethering imposed by the power cord needed for the high volume pump.

### Tripod

For stationary air monitors, a tripod or other similar device is required to hold the filter cassette at a specified elevation above the floor. As noted below, this will typically be a height that represents the breathing zone (1.5-2 meters).

### Spring Clips

For personal air monitors, the filter cassette is held in place using spring clips or other similar devices.

### Rotameter

A rotameter that has been calibrated to a primary calibration source is required to calibrate the air flow rate at the start and the end of each sampling period. Due to its dependency on changes in atmospheric pressure, the rotameter must be calibrated to a primary calibration source at the site location (e.g., City of Libby) prior to sampling and re-calibrated on-site every week. Record calibration and re-calibration to the primary standard in the field logbook.

### Primary Calibration Source

A bubble buret or other primary calibration standard may be used to calibrate the rotameter.

### Sample Labels

A pre-printed sheet of sample labels (2 identical labels per sample number) is required. One label should be attached to the filter cassette before the sample collection period begins, and the matching label should be attached to the field data sheet that records relevant data on the sample being collected.

### Field Log Book

A field log book is required to record relevant information regarding the collection of samples (location, time, unusual conditions or problems, etc.).

### Field Data Sheet



A personal air or stationary air monitoring field data sheet (as appropriate) is required to record the relevant sampling information. Refer to the Phase 2 QAPP (EPA, March 2001) for the form.

### 8.0 Instrument Calibration

External calibration devices such as a bubble buret or a rotometer that have been calibrated to a primary calibration source may be used to calibrate air flow rate prior to air sampling. The flow rate must also be measured by the same method at the end of the sampling period.

#### 8.1 Calibrating a Rotameter with an Electronic Calibrator (DryCal)

- See manufacturer's manual for operational instructions.
- To set up the calibration train, attach one end of the tygon tubing to the outlet plug of the rotameter; attach the other end of the tubing to the inlet plug on the pump. Another piece of tubing is attached from the inlet plug of the rotameter to the outlet plug on the DryCal.
- Rest or firmly stabilize the rotameter so that it is vertical ( $\pm 6^\circ$ ).
- Attach an isolating load with a pressure drop of about 10 to 20 inches of water column in series with a stable pump (a filter cassette of same lot number as will be used for field samples works well for this).
- Turn the DryCal and sampling pump on.
- Turn the flow adjust screw (or knob) on the pump until the desired flow rate is attained.
- Record the DryCal flow rate reading and the corresponding rotameter reading in the field logbook. The rotameter should be able to work within the desired flow range.
- Perform the calibration three times until the desired flow rate of  $\pm 5\%$  is attained. Once at the sampling location, a secondary calibrator (e.g., rotameter) may be used to calibrate sampling pumps.

#### 8.2 Calibrating an Air Pump with a Rotameter

A rotameter can be used provided it has been precalibrated to a primary calibration source at the site location (e.g., City of Libby). Three separate constant flow calibration readings should be obtained both before sampling and after sampling. The mean value of these flow rate measurements shall be used to calculate the total air volume sampled.

Turn on the sampling pump and run for 5 minutes before performing calibration.

- Remove the end plugs on the filter cassette. A cassette, representative of the lot planned for use in air sampling, must be used.
- To set up the calibration train, attach one end of the tygon tubing to the cassette base; attach the other end of the tubing to the inlet plug on the pump. Another piece of tubing is attached from the cassette cap to the rotameter.

- Rest or firmly stabilize the flow meter so that it is vertical ( $\pm 6^\circ$ ).
- Turn the flow adjust screw (or knob) on the sampling pump until the center of the float ball on the rotameter meets the flow rate value specified in the project plan.

## 9.0 Sample Collection

Apply one of the pre-printed adhesive labels to the filter cassette and apply the other to the field data sheet for the sample.

Secure the filter cassette in the appropriate sampling location. For a fixed air monitor, this will generally be at a height that represents the breathing zone of the potentially exposed population (e.g., 1.5- 2 meters above the floor). For personal air monitoring, the cassette will typically be placed on the lapel just below the face of the individual being monitored. For personal air sampling for Scenarios 2 and 3 [Refer to Phase 2 QAPP (EPA March 2001)], secure the cassette on the lapel of the dominant hand of the worker. The distance from the nose/mouth of the person to the cassette should be about 10 cm. Secure the cassette on the collar or lapel using spring clips or other similar devices. In all cases, orient the cassette so the open face of the cowl is pointing downward to avoid any particles entering the filter by precipitation. Remove the protective cap over the open face of the cowl and turn on the calibrated pump. Record the starting time, the initial flow rate, and all other relevant sample data on the field data sheet for the sample. Store covers and end plugs in a clean area (e.g., a closed bag or box) during the sampling period.

For sampling events lasting longer than 2 hours, in-field pump checks should be performed approximately every 2 hours. These periodic checks should include the following activities:

- Observe the sampling apparatus (filter cassette, pump, tripod, etc.) to determine whether it's been disturbed.
- Check the pump to ensure it is working properly and the flow rate is stable at the prescribed flow rate.
- Inspect the filter for overloading and particle deposition. Inspect the filter using a small flashlight. Look for particle adhesion or deposition on the side of the cassette and check the filter surface for accumulation of visible dust or smoke particles. If particle deposition on the inside of the cowl is observed, it may be necessary to ground the cowl to reduce static charge.

After the specified sampling period has elapsed, measure the ending flow rate and ending clock time on the data sheet. Turn off the pump and remove the cassette from the pump. Attach and secure a sample seal around each sample cassette in such a way as to assure that the end cap and

base plug cannot be removed without destroying the seal. Tape the ends of the seal together since the seal is not long enough to be wrapped end-to-end. Initial and date the seal.

#### 10. Sample Handling and Preservation

Package the cassettes so they will not rattle during shipment nor be exposed to static electricity. Place custody seals, dated and marked with the packager's signature, onto the shipping container. Do not ship samples in polystyrene peanuts, vermiculite, paper shreds, or excelsior. Tape sample cassettes to sheet bubbles and place in a container that will cushion the samples in such a manner that they will not rattle. For additional shipping requirements, see the project plan.

Ship the sealed cassette to the analytical laboratory under proper chain of custody procedures. No preservation of the cassette is required.

### QUALITY CONTROL and QUALITY ASSURANCE

#### Pre-Project Filter ("Lot") Blanks

Before samples are collected, two cassettes from each filter lot of 100 cassettes should be randomly selected and submitted for analysis. The lot blanks will be analyzed for asbestos fibers by the same method as will be used for field samples. The entire batch of cassettes should be rejected if any asbestos fiber is detected on any filter.

#### Field Blanks

Blank samples are used to determine if any contamination has occurred during sample handling. Prepare two blanks (from the sample lot used for field sampling) for the first 1 to 20 samples. For sets containing greater than 20 samples, prepare blanks as 10% of the samples. Filter blanks should be taken to a sampling location and prepared there. Remove the caps on the filter cassette and hold the cassette open for about 30 seconds. Close and seal the cassette as described in Section 9. Store blanks for shipment with the sample cassettes.

## REFERENCES

NIOSH 7400

NIOSH 7402

ISO 10312

OSHA Technical Manual

EPA SOP 2015

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**ATTACHMENT 2**

**LIBBY OU3 FIELD SAMPLE DATA SHEET (FSDS)  
SMOKE MONITORING STATIONARY AIR MONITOR**

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Station ID: \_\_\_\_\_ Field Logbook No: \_\_\_\_\_ Page No: \_\_\_\_\_

Station Comments: \_\_\_\_\_

X coord: \_\_\_\_\_ Y coord: \_\_\_\_\_ Elevation: \_\_\_\_\_ m

Data Item	Cassette 1	Cassette 2	Cassette 3	Cassette 4
Index ID	[affix label here]	[affix label here]	[affix label here]	[affix label here]
Field QC Type (circle one)	FS-(field sample)    FB-(field blank)	FS-(field sample)    FB-(field blank)	FS-(field sample)    FB-(field blank)	FS-(field sample)    FB-(field blank)
Pump ID Number				
Flow Meter ID Number				
Start Date (mm/dd/yy)				
Start Time (hh:mm)				
Start Flow (L/min)				
Stop Date (mm/dd/yy)				
Stop Time (hh:mm)				
Stop Flow (L/min)				
Pump fault? (circle one)	Yes                  No	Yes                  No	Yes                  No	Yes                  No
Sample Air Volume (L)				
Field Comments				
Cassette Lot Number: _____				

For Data Entry Completion (Provide Initials)	Completed by:	QC by:
----------------------------------------------	---------------	--------



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**ATTACHMENT 3**

**VOLUME CALCULATOR SPREADSHEET TOOL**  
*[provided electronically]*

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